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COMMITTEE ON PAPERS AND PUBLICATIONS.

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PAPERS ACCEPTED FOR PUBLICATION:

The Proportions of Chlorine and of Nitrogen as Nitric Acid and as Ammonia in Certain Tropical Rain-Waters. By J. B. Harrison and John Williams.

Improvements in the Colorimetric Test for Copper. By George L. Heath.

The Oxalates of Zirconium. By F. P. Venable and Charles Baskerville.

Preparation of Sodium Nitroprusside. By F. S. Hyde.

Mechanical Analysis of Basic Phosphatic Slag. By H. W. Wiley.

Analysis of an Iron Rail taken from a Gallery of an Unused Coal Mine. By H. P. Talbot and A. G. Woodman.

Estimation of Boric Acid in Foods. By L. de Koningh.

Issued with December Number, 1896.

Proceedings.

COUNCIL.

At the Buffalo meeting of the American Chemical Society it was voted that the Council be requested to take into consideration ways and means for bringing the Summer meeting of the Society into closer relation with that of Section C of the A. A. A. S., so that both meetings, if possible, may be held within the same week, thus affording the opportunity for all chemists to attend both meetings.

Inasmuch as both these bodies were well represented at the meeting referred to, it was suggested that a good deal of time could be gained by appointing then and there a committee of conference from each. This was accordingly done, the committee on the part of the American Chemical Society being Messrs. W. P. Mason, W. McMurtrie, Edward Hart, T. H. Norton and A. B. Prescott.

A joint meeting of this Committee was held with a Committee of Section C, and the following recommendations were agreed upon :

1st. Section C to have a business meeting for purposes of organization on Monday of the week of meeting, and the Vice-President's address to take place late in the afternoon of that day.

2nd. The American Chemical Society to be given Monday and Tuesday for their work.

3rd. Section C of the A. A. A. S. to be given the balance of the week.

4th. The arrangement of the program for the reading of papers before the two bodies to be left to the discretion of the President of the American Chemical Society and the Vice-President of Section C of the A. A. A. S.

These recommendations were approved by Council Oct. 27, 1896.

In view of the increasing number of papers presented at the meetings, the Council has decided that the Troy meeting shall extend over three days if this shall be found necessary.

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MEETINGS OF THE SECTIONS.

RHODE ISLAND SECTION.

A meeting of the Rhode Island Section was held at Providence, on Thursday evening, October 29, 1896.

Mr. E. D. Pearce mentioned the results of his experiments in bleaching brown tower acid. Samples of acid taken before and after bleaching were exhibited. Mr. Pearce also stated that the coloring matter of the anthers of the wild evening primrose was altered by acids and alkalies in the same way as turmeric.

A paper was read by W. M. Saunders upon "The Determination of Sulphur in Iron." The reader described briefly the deleterious effect of sulphur in iron. The small amount permitted in foundry work, and the difficulty of determining this amount was mentioned.

Next a description of methods of analysis was given. The reader considered the evolution methods, although not in every case giving the full sulphur contents of the iron, to be accurate enough for practical purposes. The results compare favorably with the oxidation method.

NEW YORK SECTION.

The November meeting of the New York Section was held on the 6th, Professor McMurtrie in the chair, and fifty-one members present.

The chair announced the acceptance by the executive committee of an invitation from Drs. Morton and Leeds to hold the December meeting at the Stevens Institute of Technology.

The death of Mr. Alfred H. Mason was announced and a sketch of his life was read.

A motion was made and seconded that the executive committee be recommended to authorize the secretary to employ a stenographer to report the discussions of papers presented at the meetings; such report, when properly edited, to be sent to the committee on papers, for publication in the Journal.

The following papers were read:

"The Volumetric Determination of Acetone," by Dr. E. R. Squibb.

"Notes on a Chemist's Trip Abroad," by C. A. Doremus.

"A New Form of Pyknometer," by J. C. Boot.

"Improvements in the Colorimetric Tests for Copper," by Geo. L. Heath.

"Note on Solubility of Bismuth Sulphide in Alkaline Sulphides," by Geo. C. Stone.

The meeting then adjourned.

CINCINNATI SECTION.

The meeting was held on November 17, in the Lloyd Library. After welcoming the Society, Prof. J. U. Lloyd read a paper entitled "Bibliography of American Pharmacy," giving a concise history of the different editions of the U. S. Pharmacopeia and its commentaries, the various dispensatories and formularies. This paper was rendered doubly interesting by the exhibition of the rare old editions of these works from the well-filled shelves of the Lloyd Library. Prof. O. W. Martin read a paper opening the discussion on the "Teaching of Elementary Chemistry." A contribution on this subject by Dr. James Lewis Howe was read by Mr. H. B. Foote. Excerpts from paper which Prof. Paul Freer presented at the summer meeting of American Association for the Advancement of Science, elicited considerable discussion by Dr. Springer, Profs. Norton, Martin and Homburg.

Dr. William H. Crane, of Cincinnati was elected a member of the Section.

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METAL SEPARATIONS BY MEANS OF HYDROCHLORIC ACID GAS.[1]

BY J. BIRD MOYER.

Received September 26, 1896.

INTRODUCTION.

THE action of gaseous haloid acids upon metallic oxides and their salts, is a field of investigation, which, though not of recent origin, has been but lately developed. It was Debray[2] who first called attention to the volatility of molybdic acid in a stream of hydrochloric acid gas, with the formation of $MoO(OH)_2Cl_2$.

E. Péchard[3] applied this and showed that molybdic acid was completely eliminated and separated from tungstic acid, by its volatility in a current of hydrochloric acid. Since that time nothing further has been done with single haloid acids, in gas form, until quite recently. Compounds have been decomposed, salts volatilized, and separations made, by means of other gases and mixtures, which may be as effective as hydrochloric acid, but are not devoid of trouble nor nearly so neat.

Smith and Oberholtzer[4] repeated and confirmed Péchard's

1 From author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Doctor of Philosophy, 1896.

2 *Compt. rend.*, 46 1098, and *Ann. Chem.* (Liebig), 108, 250.

3 *Compt. rend.*, 114, 173.

4 *J. Am. Chem. Soc.*, 15, 1.

work in regard to the separation of molybdic acid from tungstic acid, and in addition showed that gaseous hydrobromic, hydriodic, and hydrofluoric acids acted similarly. Later, Smith and Maas[1] made use of the volatilization of molybdic acid for a close atomic mass determination of molybdenum.

Smith and Hibbs[2] showed that vanadium behaved like molybdenum. Hydrochloric acid gas completely eliminates vanadic acid from sodium vanadate. A little later they investigated the action of hydrochloric acid upon the members of Group V of the periodic system.[3]

The sodium salts of nitric, pyrophosphoric, pyroarsenic and pyroantimonic acids were used. They found nitrogen, arsenic, and antimony to be volatile in gaseous hydrochloric acid, and made it the basis of a separation of phosphoric acid from nitric acid. Lead arsenate changed completely to chloride, the arsenic being volatilized, thus affording a good quick separation. Smith and Meyer[4] tried the action of all the haloid acids upon the elements of Group V of the periodic system. They worked with sodium salts and observed: I. That nitrogen was expelled completely by all the haloid acids. II. That phosphoric acid was not acted upon. III. That arsenic acid was fully expelled by hydrochloric, hydrobromic, and hydriodic acids, but only partially by hydrofluoric acid. IV. That antimony was completely volatilized by hydrochloric acid. There was no work done on bismuth. V. Vanadium went over completely in hydrochloric acid, but only partially in hydrobromic and hydrofluoric acids. VI. Columbium forms volatile products with hydrochloric and hydrobromic acids. No knowledge of didymium was obtained. VII. Tantalum is only slightly volatile in hydrochloric acid.

P. Jannasch and F. Schmidt[5] repeated some of the work of Smith and Hibbs, in which they confirmed the separation of arsenic from lead. They anticipated a slight portion of my work, and in addition separated arsenic acid from iron, tin from

[1] *Ztschr. anorg. Chem.*, 5, 280.
[2] *J. Am. Chem. Soc.*, 16, 578.
[3] *Ibid.*, 17, 682.
[4] *Ibid.*, 17, 735.
[5] *Ztschr. anorg. Chem.*, 9, 274.

lead, tin from copper, and tin from iron, in a stream of hydrochloric acid gas.

The position of bismuth in the periodic system makes it natural to suppose that it too will be volatile in hydrochloric acid gas. This I have shown to be true, and was thus enabled to separate it from lead and copper. The action of hydrobromic acid on bismuth trioxide was also tried; it formed the bromide and then volatilized. It requires a higher temperature and longer action than with hydrochloric acid. Because of lack of time, I have been compelled to abandon the experiments instituted with a view of affecting separations, in atmospheres of hydrobromic acid and hydriodic acid gas and have confined my labors to hydrochloric acid gas.

METHOD OF WORK.

The hydrochloric acid gas was generated by dropping concentrated sulphuric acid from a separatory funnel, upon concentrated hydrochloric acid contained in a three liter flask. The gas evolved at the ordinary temperature was dried by passing it through two sulphuric acid drying bottles and then through a calcium chloride tower, when it was considered sufficiently dry for the purpose. The substance to be acted upon was weighed out in a porcelain boat and the latter was placed in a combustion tube of hard glass.

The tube had previously been rinsed with alcohol and then with ether, to remove all moisture. The ether was removed by drawing a current of dry air through the tube. This tube was connected to a two-necked bulb receiver containing about 300 cc. of distilled water. When working with arsenic ten cc. of nitric acid were added. The connecting tube from the combustion tube to the bulb receiver was made to enter the receiver and dip below the surface of the water, thus catching all volatile products, as well as taking up the hydrochloric acid gas. To insure safety from the loss of volatile products, a small flask containing water was attached to the bulb receiver. The apparatus was controlled at both ends by stop-cocks. This is necessary to prevent backward suction on disconnecting the apparatus. After the reaction was completed the boat was removed to

a sulphuric acid desiccator from which the air could be exhausted. In general, the procedure was similar to that employed by Hibbs.[1]

I.—BEHAVIOR OF ANTIMONY TRIOXIDE.

Antimony oxide, labelled chemically pure, was dissolved in hydrochloric acid and precipitated with a large amount of water. After washing by decantation it was redissolved and reprecipitated. This procedure was repeated several times, when it was precipitated by ammonium carbonate, washed, and ignited. The pure oxide obtained in this manner was subjected to the action of hydrochloric acid gas and it was found to volatilize completely. In each trial a one-tenth gram of the oxide was acted upon. The temperature varied between 150° and 190° C. It was determined in the following way: The combustion tube was slipped through two holes made in the sides of a copper drying oven.

A very slow current of gas was used as the antimony seemed to volatilize more readily and completely, if the current was slow and the heat gentle. This I attribute, on reflection, to the fact that I ignited the oxide too strongly, (to a red heat) in its preparation. It dissolved with difficulty in concentrated hydrochloric acid. Lack of time prevented the repetition of this experiment and the separation of antimony from lead and copper, in which this substance was used. About eight hours was the time required for the volatilization; very probably a shorter time would be required if the oxide had been obtained by gentle ignition.

II.—BEHAVIOR OF LEAD OXIDE.

Pure lead oxide was obtained from recrystallized nitrate, by careful ignition. This oxide changed completely into chloride at the ordinary temperature and it was only necessary to apply a gentle heat to complete the change and entirely remove the water formed. No volatilization was noticed until a temperature of 225° was reached; at this point the lead chloride slightly volatilized.

I think it possible to estimate lead as chloride, if the temperature is kept under 200°. A weighed amount of lead oxide was

[1] Thesis, 1896.

acted upon by hydrochloric acid gas in the cold, for two hours, and then heated sufficiently to remove all the water formed.

The boat was cooled in the gas, and then placed in a sulphuric acid desiccator and allowed to stand one-half hour. It was then weighed.

EXPERIMENTS.

	Lead oxide taken.	Lead chloride obtained.	Lead chloride required.	Difference.
	Gram.	Gram.	Gram.	Gram.
Experiment I	0.1017	0.1267	0.1267	0.0000
" II	0.1015	0.1258	0.1265	—0.0007
" III.....	0.1169	0.1454	0.1447	+0.0007

The lead chloride dissolved in hot water without residue.

III.—THE SEPARATION OF ANTIMONY FROM LEAD.

The oxides were carefully weighed and thoroughly mixed in a porcelain boat. Hydrochloric acid gas was passed over them in the cold, until the lead oxide had been entirely changed to the chloride. It was then heated with the smallest flame obtainable from a fish-tail burner, placed about two inches below the tube.

	Antimony trichloride taken.	Lead chloride taken.	Lead chloride obtained.	Lead chloride required.
	Gram.	Gram.	Gram.	Gram.
Experiment I	0.1015	0.1189	0.1470	0.1482
" II....	0.1090	0.1021	0.1266	0.1272
" III...	0.1350	0.0852	0.1057	0.1062
" IV ...	0.1250	0.1671	0.2069	0.2083

The time required was seven hours. The lead chloride was immediately weighed. It dissolved completely in hot water and this solution was tested by means of Marsh's apparatus for antimony, without finding the latter present. Experiment II was slightly varied by first moistening the oxides with a drop of hydrochloric acid.

IV.—BEHAVIOR OF BISMUTH OXIDE.

Bismuth nitrate, as pure as could be obtained, was dissolved in nitric acid and then thrown down with a large quantity of water. The precipitate was carefully washed by decantation. This operation was repeated several times.

It was then dissolved in acidulated water and precipitated

with ammonium hydroxide and ammonium carbonate. This, on ignition, gave pure oxide, which, heated in a stream of hydrochloric acid gas, completely volatilized as chloride. Here the same treatment is necessary as obtained for antimony. A slow current of gas and a low heat were best adapted for the volatilization (a temperature of 130°, or roughly, the heat afforded by a fish-tail burner placed two inches below the combustion tube, with a flame an eighth of an inch high). The bismuth chloride sublimed nicely, forming a white crystalline mass beyond the boat, which could be readily driven along by a gentle heat.

V.—THE SEPARATION OF BISMUTH FROM LEAD.

The same material was used as in the preceding experiments. The weighed oxides were thoroughly mixed in a porcelain boat. Usually the gas was allowed to act in the cold for an hour, which changed the oxides completely to chlorides.

The same conditions prevailed as under bismuth oxide alone. If an attempt was made to hasten the reaction by heating higher than 180°, a little lead would volatilize. This sublimate, slightly yellow in color, would appear directly over the boat and could not be driven along the tube like bismuth, hence it was readily detected.

The separation of bismuth from lead requires much care, as it is not as sharp as could be desired. It is also difficult to tell exactly when the last traces of bismuth have been driven out of the boat, as there was no color change to indicate it, both metals forming white chlorides. The separation is complete in from six to seven hours. At the end of the separation the position of the boat was changed and the action continued; if no further sublimation occurred it was cooled and removed to a desiccator. The weight was taken after standing one-half hour over sulphuric acid. With care bismuth can be separated from lead in this manner.

	Lead oxide taken. Gram.	Bismuth trioxide taken. Gram.	Lead chloride obtained. Gram.	Lead chloride required. Gram.	Difference. Gram.
Experiment I	0.1014	0.2020	0.1261	0.1264	—0.0003
" II....	0.1006	0.0642	0.1252	0.1254	—0.0002
" III...	0.1038	0.1003	0.1294	0.1302	—0.0008
" IV ...	0.1412	0.1260	0.1759	0.1759	+0.0000

The chloride of lead dissolved completely in hot water. It showed no bismuth. The sublimate contained no lead.

VI.—BEHAVIOR OF CUPRIC OXIDE.

Pure copper nitrate was made by recrystallization. It was then ignited in a porcelain crucible at a dull red heat, until it became constant in weight. The pure black oxide was then subjected to the action of hydrochloric acid gas. In Experiment I, the boat containing the oxide was heated at the outset to 175°. It was taken out after two hours, placed over sulphuric acid for half an hour, and weighed. The weight showed that the copper oxide had hardly been acted upon. It had only been superficially changed to chloride. It was then moistened with two or three drops of hydrochloric acid, dried in a rapid current of the gas, and heated two hours longer. This resulted in the complete transformation into chloride. The anhydrous chloride thus obtained, liver brown in color, was placed in a desiccator from which the air was exhausted. This was done to remove all the gas that might be retained and prevented a too rapid absorption of moisture.

Copper chloride absorbs moisture but not so rapidly as to prevent weighing in this form:

	Copper oxide taken.	Copper chloride obtained.	Copper chloride required.	Difference.
	Gram.	Gram.	Gram.	Gram.
Experiment I	0.1011	0.1708	0.1713	—0.0005
" II....	0.1025	0.1726	0.1736	—0.0010
" III...	0.1034	0.1756	0.1752	+0.0004

In Experiment II, the change was completed in the cold by prolonged action through four hours. It was then heated about ten minutes at the end to drive out the moisture that had formed. In all the experiments cited, the copper chloride, after weighing, was found to dissolve completely in cold water.

VII.—THE SEPARATION OF ANTIMONY FROM COPPER.

The same material was used as in the preceding experiments. The weighed oxides were thoroughly mixed. The antimony was completely volatilized, leaving copper chloride which was

weighed as such. The volatile antimony chloride was caught in the bulb receiver at the end of the tube. The bulb and tube were washed out with acidulated water into a beaker and the antimony thrown down with hydrogen sulphide. The antimony sulphide was filtered, thoroughly washed, and while moist dissolved in strong hydrochloric acid. The hydrogen sulphide evolved was conducted into bromine water and oxidized to sulphuric acid, which was estimated as usual and the antimony calculated.

The length of time required was eight hours. On several occasions the experiment was interrupted at the end of four hours, but invariably the separation was incomplete and on dissolving out the copper chloride formed, black copper oxide and white antimony oxide were plainly evident. In some cases the mixture of oxides was moistened with a couple of drops of hydrochloric acid and then evaporated down in a stream of acid gas, by heating the tube over a water-bath. This treatment seemed to facilitate matters but it is not altogether advisable, because the copper chloride has a tendency to creep over the sides of the boat. It is quicker in the end to separate them in the dry condition, allowing plenty of time for the reaction. The copper chloride obtained was perfectly soluble in cold water and contained no antimony. It could readily be changed to oxide and weighed if thought necessary.

	Antimony trioxide taken.	Copper oxide taken.	Copper chloride obtained.	Copper chloride required.	Difference.
	Gram.	Gram.	Gram.	Gram.	Gram.
Experiment I ..	0.1068	0.1040	0.1750	0.1745	+0.0005
" II..	0.1062	0.1053	0.1774	0.1784	—0.0010
" III.	0.1022	0.1020	0.1726	0.1728	—0.0002
" IV .	0.1198	0.1020	0.1722	0.1728	—0.0006

	Antimony trioxide taken.	Antimony trioxide found.	Difference.
	Gram.	Gram.	Gram.
Experiment I.....	0.1068	0.1059	+0.0009

VIII.—THE SEPARATION OF BISMUTH FROM COPPER.

The pure oxides were mixed and treated as directed under bismuth and lead.

	Copper oxide taken.	Bismuth trichloride taken.	Copper chloride obtained.	Copper chloride required.	Difference.
	Gram.	Grams.	Grams.	Grams.	Grams.
Experiment I ..	0.1030	0.1069	0.1738	0.1745	—0.0007
" II..	0.1004	0.1077	0.1701	0.1713	—0.0012
" III.	0.1026	0.1060	0.1741	0.1738	+0.0003
" IV .	0.1019	0.1058	0.1718	0.1726	—0.0008

	Bismuth trioxide obtained.	Bismuth trioxide required.	Difference
	Gram.	Gram.	Gram.
Experiment I.....	0.1076	0.1069	+0.0007

The time required in each of these trials was seven hours. It seemed to be advantageous to raise the temperature and heat sharply for about ten minutes at the end, to insure the complete removal of the bismuth.

Moistening with acid helped the reaction but subjected it to the same danger of creeping as noted under antimony and copper.

The bismuth was estimated as follows: It was washed out of the tube and bulb with acidulated water and then precipitated as sulphide. The bismuth sulphide was filtered, washed, and dissolved in nitric acid. It was thrown out of the solution with ammonium hydroxide and ammonium carbonate, as hydrated oxide, and then filtered, dried, and ignited. It was weighed as oxide. The residue of copper chloride in the boat dissolved in cold water and showed no bismuth.

IX.—ACTION OF GASEOUS HYDROCHLORIC ACID ON SODIUM PYROARSENATE.

Hibbs[1] showed that arsenic was completely volatilized from sodium pyroarsenate, leaving weighable sodium chloride. In fact, so clean was the elimination of arsenic that he made it the basis of an arsenic atomic mass determination, with admirable success.

In working up the separation of arsenic from other metals it was necessary to start with the pure sodium salt. After purification I decided to test it, by weighing the salt produced by the action of the acid gas upon it. Several determinations gave close results, proving the salt pure.

[1] See next paper, page 1044.

Chemically pure arsenate was procured. It was recrystallized and then ignited (not too strongly) for an hour. The pyroarsenate obtained was used in precipitating the various arsenates investigated.

	Sodium pyroarsenate taken.	Sodium chloride obtained.	Sodium chloride required.
	Gram.	Gram.	Gram.
Experiment I.....	0.2021	0.1330	0.1335
" II	0.1039	0.0691	0.0686

X.—THE SEPARATION OF ARSENIC FROM COPPER.

Pure sodium pyroarsenate was used to precipitate the copper salt.

Copper sulphate was recrystallized five times, a few good crystals were dissolved and the two solutions mixed. A green copper arsenate was precipitated. It was washed and dried at 100°. Salkowski[1] observes that copper arsenate still contains water above 130°. My salt had the composition $Cu_3As_2O_8 + 2H_2O$.

Hydrochloric acid gas completely changes it in the cold to chloride. A slight heat drives out the arsenic and water and leaves a brown anhydrous copper chloride, which can be weighed as such. Care was taken to remove all the acid gas before weighing.

The arsenic was washed out of the bulb into a beaker, this was warmed with nitric acid to insure oxidation, and then it was precipitated from an ammoniacal solution with "a magnesia mixture." It was weighed as $Mg_2As_2O_7$.

	Copper arsenate taken.	Copper chloride obtained.	Copper chloride required.	Difference.
	Gram.	Gram.	Gram.	Gram.
Experiment I	0.1067	0.0850	0.0851	—0.0001
" II....	0.1240	0.0998	0.0991	+0.0007
" III...	0.1072	0.0860	0.0856	+0.0004
" IV ...	0.1155	0.0924	0.0923	+0.0001
" V	0.1042	0.0832	0.0833	—0.0001

Experiment I. As_2O_5 obtained, 0.0498 gram; As_2O_5 required, 0.0487 gram.

The residue of copper chloride completely dissolved in water. It showed no arsenic when tested in a Marsh apparatus.

[1] *J. prakt. Chem.*, **104**, 129.

XI.—THE SEPARATION OF ARSENIC FROM SILVER.

Silver arsenate was made by precipitating silver nitrate with sodium arsenate. Care was taken to have the nitrate in excess. The reddish-brown arsenate of silver was washed with boiling water, until the washings no longer showed silver, when tested with hydrochloric acid. It was dried at 110°.

As was expected, the acid gas attacked it even in the cold. In fact the action was so vigorous that a couple of analyses were spoiled by spattering. The trouble arose from the fact that the arsenate was not finely powdered. Heat was generated in the reaction sufficiently to send over a portion of the water formed. Experiment I was run in the cold for one hour and then heated sharply, for a few minutes, to expel the arsenic and water. The result was only 0.46 per cent. too high, but indicated that the salt should be heated longer, and not necessarily as high to remove all the arsenic.

The succeeding experiments were heated from one to two hours at 150° with better results:

	Silver arsenate taken.	Silver chloride obtained.	Silver chloride required.	Difference.
	Gram.	Gram.	Gram.	Gram.
Experiment I.....	0.2542	0.2381	0.2363	+0.0018
" II....	0.2325	0.2163	0.2161	+0.0002
" III...	0.2084	0.1952	0.1938	+0.0014
" IV ...	0.2070	0.1927	0.1924	+0.0003

Experiment I. Ag obtained = 70.45 per cent; Ag required = 69.99 per cent.

The residues in Experiments II, III, and IV were dissolved and tested for arsenic. None was found.

XII.—THE SEPARATION OF ARSENIC FROM CADMIUM.

Chemically pure cadmium sulphate was precipitated by a solution of sodium pyroarsenate. Stirring brought out a gelatinous arsenate, which changed by additional stirring to a granular salt. This was thoroughly washed and dried at 110°. It had the composition $Cd_2As_2O_7 + 2H_2O$. Salkowski[1] observes that a red heat is necessary to fully dehydrate this salt.

The moisture and arsenic were completely expelled at 150°,

[1] *Loc. cit.*

leaving a uniform mass of cadmium chloride. It was weighed as such after standing over sulphuric acid for one-half hour. The arsenic was determined as usual.

	$Cd_3As_2O_8 + 2H_2O$ taken.	Cadmium chloride obtained.	Cadmium chloride required.	Difference.
	Gram.	Gram.	Gram.	Gram.
Experiment I	0.2359	0.1965	0.1977	—0.0012
" II ...	0.1166	0.0968	0.0968	—0.0000
" III...	0.1030	0.0857	0.0855	+0.0002
" IV ...	0.1138	0.0947	0.0946	+0.0001
" V	0.1043	0.0870	0.0867	+0.0003

	$Cd_3As_2O_8 + 2H_2O$ taken.	As_2O_5 obtained.	As_2O_5 required.	Difference.
	Gram.	Gram.	Gram.	Gram.
Experiment I	0.2359	0.0813	0.0822	0.0009

The cadmium chloride dissolved perfectly in water and showed no arsenic, when tested in a Marsh apparatus.

XIII.—THE ACTION OF HYDROCHLORIC ACID GAS ON FERRIC OXIDE.

Pure oxide of iron was heated in a stream of acid gas. The behavior of iron is rather peculiar, as it very readily changes into chloride, and then only partially volatilizes. On heating to 200° the greater part is driven over as flaky crystals of ferric chloride. The remainder consists of a white mass, which refuses to go over on prolonged action and also on raising the temperature.

This residue was soluble in water and did not react with potassium thiocyanate, but immediately gave a blue precipitate with ferricyanide. Reduction was therefore evident; this is also noted by Jannasch and Schmidt.[1] The temperature at which ferric chloride usually goes into the ferrous condition is above 1000°.

Care was taken to prepare perfectly pure hydrochloric acid gas. Chemically pure acids were used to this end. The action however was the same in all cases.

XIV.—THE SEPARATION OF ARSENIC FROM IRON.

Chemically pure ferrous ammonium sulphate was carefully

[1] *Loc. cit.*

oxidized with nitric acid, it was taken up in water, filtered and then crystallized several times. The best crystals were selected and a solution made to precipitate the arsenate. A white precipitate tinged with yellow was formed. It was washed by decantation and then filtered and washed until the washings no longer gave Prussian blue with ferrocyanide. It was then dried and gently ignited.

The acid gas acts on it quickly in the cold and it becomes a light green liquid. In evaporating off the moisture the chloride of iron was carried over with the arsenic.

In a second trial, with the temperature lower and occasionally removing the source of the heat altogether, when ebullition threatened to cause spattering, ferric chloride was obtained without loss. This was gradually heated a little higher to remove all the arsenic.

The chloride of iron was dissolved, oxidized, precipitated with ammonium hydroxide and estimated as usual. The result was fair and the product tested showed the absence of arsenic, but all succeeding experiments failed. Either the substance spattered or the iron went along with the arsenic.

Jannasch and Schmidt[1] separated arsenic from iron by placing their material in a large hard glass bulb and evaporating down to dryness with nitric acid, in an air current. This is not applicable when a porcelain boat is employed. They then volatilized the arsenic in hydrochloric acid gas at 120°.

XV.—SEPARATION OF ARSENIC FROM ZINC.

In some preliminary work zinc oxide was treated with acid gas at 200°. It completely changed to chloride and was not volatile. Pure zinc sulphate was used to precipitate the arsenate; it was washed, dried and ignited to 150°. The same difficulty appeared as was encountered under iron. Zinc arsenate melts down to a liquid mass as soon as the acid gas strikes it, which is extremely hard to evaporate without spattering. A small glass cover was placed over the boat, which tended to lessen the spattering, but did not entirely prevent it.

The zinc was estimated by taking the chloride up in a little

[1] *Loc. cit.*

hydrochloric acid and running it down with pure mercuric oxide. It was then ignited and weighed as zinc oxide. One good result was obtained, but generally the residues of zinc contained arsenic and the results were far from being concordant.

XVI.—THE SEPARATION OF ARSENIC FROM COBALT AND NICKEL.

Cobalt and nickel were precipitated as arsenates in the usual manner, with a solution of pyroarsenate.

Cobalt nitrate, a Merck preparation, was carefully purified; considerable manganese was found and eliminated.

This gave the pink salt $Co_3As_2O_8 + 8H_2O$, which was ignited to the blue anhydrous compound.

Cobalt arsenate is very readily attacked by the acid gas in the cold, yielding a pink chloride. A slight heat, not much above 120°, changed it to the blue chloride and drove out the arsenic. At first it was quickly weighed as chloride, then it was taken up in a little hydrochloric acid and evaporated down with mercuric oxide. On ignition, black Co_3O_4 was obtained and weighed.

The arsenic was estimated as usual.

	Experiment I. Gram.	Experiment II. Gram.
$Co_3As_2O_8$ taken	0.1509	0.2029
$CoCl_2$ obtained	0.1309	....
$CoCl_2$ required	0.1293	....
Co_3O_4 obtained	0.0738	0.0969
Co_3O_4 required	0.0731	0.0983
Difference	+0.0007	—0.0014
As_2O_5 obtained	0.0770	....
As_2O_5 required	0.0764	....
Difference	+0.0006	....

On testing the cobalt residue by the Marsh test, no trace of arsenic was found. No cobalt was found in the sublimate. Some of the first experiments gave cobalt too low; it was thought that they had been heated too high, but testing showed no volatilized cobalt.

A temperature of 125° is sufficient to drive out all of the arsenic, and at this temperature there is no danger of volatilizing the cobalt.

In working with nickel, the green arsenate was simply dried

in the first experiment. It therefore had the composition $Ni_3As_2O_8 + 8H_2O$.

Hydrochloric acid gas attacked it in the cold. A slight heat drives out the arsenic and moisture and leaves a salmon-colored chloride. The nickel chloride was changed to oxide by evaporating it with nitric acid and igniting.

	Experiment I. Gram.
$Ni_3As_2O_8 + 8H_2O$ taken	0.1502
NiO obtained	0.0554
NiO required	0.0561
Difference	−0.0007

In Experiments II and III the salt was made anhydrous by ignition.

	Experiment II. Gram.	Experiment III. Gram.
$Ni_3As_2O_8$ taken	0.1166	0.1040
NiO obtained	0.0577	0.0523
NiO required	0.0575	0.0513
Difference	+0.0002	+0.0010
As_2O_5 obtained	....	0.0515
As_2O_5 required	....	0.0526
Difference	....	−0.0011

The Marsh test showed no arsenic with the nickel.

XVII.—BEHAVIOR OF MINERALS IN HYDROCHLORIC ACID GAS.

Niccolite. One-half gram of the mineral was finely powdered and subjected to the action of acid gas for a day, at a temperature of 200° C. It was only very slightly affected.

A second portion was dissolved in nitric acid and evaporated down in a porcelain dish. It was then transferred to a boat and evaporated to dryness. To remove all the acid, it was heated in an oven to 110° for one-half hour. The dry substance was acted upon by the acid gas in the cold for five hours. It changed completely to chloride. A temperature of 150° for an hour removed all the moisture and arsenic.

The nickel chloride was evaporated down with nitric acid, ignited, and weighed as NiO. The arsenic was estimated as usual.

	Per cent.
Nickel found	43.79
Nickel calculated	43.60
Difference	0.19
Arsenic found	56.66
Arsenic calculated	56.40
Difference	0.26

Undoubtedly there is still a wide field open in regard to the behavior of hydrochloric acid gas upon mineral species. Smith and Hibbs[1] showed that mimetite lost its arsenic quantitatively, when heated in a stream of acid gas. In this laboratory others are being investigated with favorable indications. The direct employment of hydrochloric acid gas upon a powdered mineral would simplify many a tedious gravimetric process, leaving the separated elements in a desirable condition for further treatment.

In the case of a mineral such as niccolite, where it must first be decomposed with nitric acid and then transferred to a boat, the advantage is not so great. This, however, can be modified, so that the time factor is reduced and the advantage of the method still retained. Instead of using a boat, which has no advantage unless the non-volatile chlorides are to be weighed directly, a hard glass bulb can be substituted. The mineral is placed in the bulb, dissolved in nitric acid, and evaporated down by the aid of a current of air drawn through the bulb.

The residual oxides are then separated in a stream of hydrochloric acid gas as usual.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, NO. 15.]

THE ATOMIC WEIGHTS OF NITROGEN AND ARSENIC.[2]

BY JOSEPH GILLINGHAM HIBBS.

Received September 26, 1896.

THE atomic weight of the metal molybdenum had been determined by expelling molybdic acid from sodium molybdate with hydrochloric acid gas, then weighing the residual sodium chloride.

[1] *Loc. cit.*

[2] From author's thesis presented to the Faculty of the University of Pennsylvania for the degree of Doctor of Philosophy, 1896.

Having found that nitric acid and arsenic acid were driven from their alkali salts with ease, leaving a chloride that was absolutely pure, and believing that the atomic masses of nitrogen and arsenic determined in this manner would afford a valuable contribution to the literature relating to these constants, a carefully conducted series of experiments was made with two nitrates and one arsenate. The results are given in detail in the following lines:

THE ATOMIC WEIGHT OF NITROGEN.

In the past, determinations of the atomic weight of nitrogen have been made from the density of the gas itself, from the ratio between ammonium chloride and silver, and from the decomposition of certain nitrates. The first method in particular has been frequently applied. Thomson, Dulong, Berzelius, and Lavoisier brought to light many new facts relating to the atomic weight of nitrogen; unfortunately, however, considerable that they have presented has been affected by complications that have introduced inaccuracies.

Dumas and Boussingault[1] found the mean density of nitrogen to be 0.972; for hydrogen they found a mean density of 0.0693, which would give nitrogen an atomic weight of 14.026. Regnault obtained a more concordant series of results, the mean being 0.97137, and a density for hydrogen of 0.0692, which makes the atomic weight of nitrogen equal to 14.0244.

Clarke gives in detail his computation of the means of the results obtained by Penny, Stas, and Marignac. Their work on the determination of the atomic weight of this particular element was mainly on the ratio of ammonium chloride and silver, and the decomposition of certain nitrates. A great degree of accuracy was maintained throughout the entire investigation; but the amount of work required to obtain a single result necessarily lays the method open to a serious error of manipulation.

In this connection a paragraph from Clarke's "A Recalculation of the Atomic Weights" may be cited: "The general method of working upon these ratios is due to Penny. Applied to the ratio between the chloride and nitrate of potassium, it is

[1] *Compt. rend.*, 1841—12. 1005.

as follows: A weighed quantity of the chloride is introduced into a flask which is placed upon its side and connected with a receiver. An excess of pure nitric acid is added, and the transformation is gradually brought about by the aid of heat, the nitrate being brought into a weighable form. The liquid in the receiver is also evaporated, and the trace of solid matter which has been mechanically carried over, is recovered and also taken into account."

The method indicated in this study, and actually applied with the results appended, is decidedly less objectionable. In this method there is no distillation, no precipitate, in fact, nothing that could involve serious error.

Clarke summarizes the results of Penny, Stas, and Marignac as follows:

1.	From specific gravity of N	N = 14.0244
2.	" ammonium chloride	N = 14.0336
3.	" ratio number four	N = 14.0330
4.	" silver nitrate	N = 13.9840
5.	" potassium nitrate	N = 13.9774
6.	" sodium nitrate	N = 13.9906
	Mean of results for N	N = 14.0210

If oxygen is 16, this becomes 14.0291. Stas found the atomic weight of nitrogen to be 14.044. Dumas found 14 by experiments on the combustion of ammonia and cyanogen (O = 16). Pelouze found 14.014 by bringing a known weight of silver nitrate in contact with a known and slightly excessive weight of ammonium chloride, which excess was titrated. Anderson found 13.95 by the decomposition of the nitrate of lead, with just enough heat for decomposition (the same method that was used by Berzelius). Marignac found 14.02 by dissolving a known weight of silver in nitric acid and then melting and weighing the nitrate found.

A.—ATOMIC WEIGHT OF NITROGEN BY ACTION OF HYDROGEN CHLORIDE UPON POTASSIUM NITRATE.

The purest salt obtainable was dissolved in water, filtered, and recrystallized six times, a solution of which was tested for chlorides, sulphates, etc., but no impurity was found. One more crystallization was made and the best crystals were

selected. These were washed with distilled water and dried at 210° C. for three hours, powdered, and again dried, and finally placed in a weighing bottle. This compound was dried before each experiment. It was also allowed to stand in a balance case one hour before weighing. The same degree of care was exercised in the preparation of the boat for weighing.

The weighing bottle was placed on the scale pan and allowed to stand several minutes in order to regain its normal temperature. After weighing it was quickly opened and a portion of the salt removed to the boat and again closed and allowed to stand in the balance case for several hours before reweighing. The boat was then introduced into the combustion tube and the gas passed over it. The characteristic action took place. The only difference in the method of procedure adopted here and that described in the first section of this paper, was a longer time being given to complete the action, using a lower temperature, in order to do away with all possibility of fusion of the salt. It was then carefully removed to a vacuum desiccator and allowed to stand over night before weighing. It may be said also that experiments were only conducted on clear days to insure the non-entrance of moisture.

With potassium nitrate, no great variation of amount was taken.

Five determinations were made in this case:

No.	Potassium nitrate taken.	Potassium chloride obtained.	Correction for potassium nitrate.	Correction for potassium chloride.	Correction for weight of potassium nitrate	Correction for weight of potassium chloride.	Molecular weight of potassium nitrate obtained.	Atomic weight of nitrogen obtained.
	Gram.	Gram.	Gram.	Gram.	Gram.	Gram.		
1.	0.11084	0.08173	0.00006	0.00004	0.11090	0.08177	0.101121	14.011
2.	0.14864	0.00960	0.00007	0.00005	0.14871	0.10965	0.101120	14.010
3.	0.21056	0.15525	0.00011	0.00008	0.21067	0.15533	0.101123	14.013
4.	0.23248	0.17214	0.00012	0.00009	0.23360	0.17223	0.101121	14.011
5.	0.24271	0.17894	0.00013	0.00009	0.24284	0.17903	0.101124	14.014

Atomic weight of nitrogen = 14.0118 ± 0.000472.

The atomic values used in these calculations were taken from "Table of Atomic Masses," revised by F. W. Clarke, in October, 1891.

The figures deduced from these values are, of course, subject to any change made by later revision of atomic weights. It is not so much the exact figure to which attention is called, as to the constancy of result brought forward by this method. The values used were:

Oxygen	16.00
Potassium	39.11
Chlorine	35.45
Specific gravity potassium nitrate	2.1
Specific gravity potassium chloride	1.99

B.—ATOMIC WEIGHT OF NITROGEN BY ACTION OF HYDROGEN CHLORIDE UPON SODIUM NITRATE.

The same degree of care and method of procedure were here observed as in Division A. The results are as follows:

No.	Potassium nitrate taken.	Sodium chloride obtained.	Correction for sodium nitrate.	Correction for sodium chloride.	Correction for sodium nitrate.	Correction for sodium chloride.	Molecular weight of sodium nitrate.	Atomic weight of nitrogen.
	Gram.	Gram.	Gram.	Gram.	Gram.	Gram.		
1.	0.01550	0.01064	....	....	0.01550	0.01066	85.061	14.011
2.	0.20967	0.14419	0.00009	0.00007	0.20976	0.14426	85.061	14.011
3.	6.26217	0.18029	0.00012	0.00009	0.26229	0.18038	85.064	14.014
4.	0.66610	0.46805	0.00035	0.00024	0.66645	0.45829	85.064	14.014
5.	0.93676	0.64422	0.00042	0.00034	0.93718	0.64456	85.058	14.008

Atomic weight of nitrogen = 14.0116 ± 0.000741.

Atomic values used were

Oxygen	16.00
Sodium	23.05
Chlorine	35.45
Specific gravity sodium chloride	2.16
Specific gravity sodium nitrate	2.26

When these results are compared with those obtained by Penny and Stas by treatment of potassium chloride with nitric acid, and the treatment of potassium nitrate with hydrochloric acid (likewise for sodium), a close comparison can be made.

Penny.	Hydrogen chloride method.
For potassium nitrate....13.9774	For potassium nitrate....14.0118
" sodium nitrate.....13.9906	" sodium nitrate.....14.0116

Showing a difference of

0.0344 for potassium salt,
0.0210 for sodium salt.

When a mean of the above results is taken, the atomic weight of nitrogen equals

13.9996 for potassium salt,
14.0011 for sodium salt.

Taking now a mean of these values, the atomic weight of nitrogen would be 14.0003.

C.—THE ATOMIC WEIGHT OF ARSENIC.

The atomic weight of arsenic has been obtained from the chloride ($AsCl_3$), the bromide ($AsBr_3$), and the trioxide (As_2O_3).

Pelouze, in 1845,[1] and Dumas, in 1859, determined it by the titration with known quantities of pure silver in the analysis of arsenic trichloride. The mean of their results, as computed by Clarke, gives the atomic weight of arsenic, 74.829. Wallace[2] makes the same titration with silver in the analysis of arsenic tribromide. His value is 74.046. Kessler made a set of determinations by estimating the amount of potassium bichromate required to oxidize 100 parts of arsenic trioxide to arsenic pentoxide. He obtained a mean value of 75.002.

A mean of these results gives the following :

From $AsCl_3$	74 829
" $AsBr_3$	74.046
" As_2O_3	75.002
General mean	74.918

If oxygen = 16, then the atomic weight of arsenic will equal 75.090.

Berzelius, in 1826, heated sulphur and arsenic trioxide together in such a way that sulphur dioxide alone escaped ; this method gave 74.840 as the atomic weight of arsenic. But one experiment was made, so that it does not possess much value. In the above method there seems to be a wide variation in the results obtained, the difference between the extreme values is but little less than one unit.

By the hydrogen chloride method, we have but the weighing of the material used in the determination—which must necessarily enter every estimation or analysis—and a single weighing after the action of the acid gas. As in the case of nitrogen, the method seems to be as short and concise as possible.

[1] *Compt. rend.*, 10, 1047.
[2] Phil. Mag. (4), 18, 279.

The methods and *modus operandi* were exactly the same as those used in the determination of the atomic weight of nitrogen.

The sodium chloride obtained was perfectly white in color. In no instance was it fused. After weighing the salt residue it showed no traces of arsenic, and was readily soluble in cold water without residue. The same conditions of atmosphere were observed.

As the specific gravity of sodium pyroarsenate could not be obtained, it was determined by means of the specific gravity bottle, against chloroform, and was found to be 2.205, while the specific gravity of sodium chloride was taken as 2.16. The atomic values used were:

Oxygen	16.00
Sodium	23.05
Chlorine	35.45

The results here obtained, besides being to a great degree constant, compare favorably with those obtained by Pelouze (74.829) and Kessler (75.002).

A coincidence may here be shown by the fact that the mean of these values gives 74.9155, while the hydrogen chloride method gives 74.9158.

In order to give the method a thorough trial, the amounts taken cover a wide range. The smallest amount used was 0.02176 gram of sodium pyroarsenate, and the largest 3.22485 grams. It will also be noticed that the variation in result is but 0.027 for ten determinations.

No.	Sodium pyroarsenate taken.	Sodium chloride obtained.	Correction for sodium pyroarsenate.	Correction for sodium chloride.	Correction for sodium pyroarsenate.	Correction for sodium chloride.	Molecular weight of sodium pyroarsenate.	Atomic weight of arsenic.
	Gram.	Gram.	Gram.	Gram.	Gram.	Gram.		
1.	0.02176	0.01439	0.00001	0.00000	0.02177	0.01439	354.008	74.904
2.	0.04711	0.03114	0.00002	0.00001	0.04713	0.03115	354.042	74.921
3.	0.05792	0.03828	0.00003	0.00002	0.05795	0.03830	354.054	74.927
4.	0.40780	0.26970	0.00021	0.00011	0.40801	0.26981	354.002	74.901
5.	0.50440	0.33028	0.00026	0.00017	0.50466	0.33045	354.033	74.916
6.	0.77497	0.51222	0.00041	0.00027	0.77538	0.51249	354.034	74.917
7.	0.82853	0.54762	0.00044	0.00029	0.82897	0.54791	354.034	74.917
8.	1.19068	0.78690	0.00056	0.00041	1.19124	0.78731	354.053	74.926
9.	1.67464	1.10681	0.00081	0.00051	1.67545	1.10732	354.057	74.928
10.	3.22485	2.13168	0.00152	0.00099	3.22637	2.13267	354.002	74.901

Atomic weight of arsenic = 74.9158 ± 0.00222.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No 16.]

THE SEPARATION OF VANADIUM FROM ARSENIC.

BY CHARLES FIELD, 3RD, AND EDGAR F. SMITH.

Received October 2, 1896.

AS vanadium and arsenic occur associated in minerals and likewise in artificial products, their separation becomes a matter of consequence.

The course usually pursued in carrying out this separation is that long since recommended for the removal of vanadic acid from its solutions; namely, its precipitation as ammonium metavanadate. Other methods have recently appeared in the literature bearing on analysis. Reference is here made especially to the publication of Fischer.[1]

Experiments made in this laboratory on the behavior of vanadates[2] and arsenates[3] heated in an atmosphere of hydrochloric acid gas, in which both acids were volatilized, suggested the thought that if the sulphides of vanadium and arsenic were exposed to the same vapors perhaps they would show a variation in deportment. And so it has proved. Perfectly dry arsenic trisulphide, previously washed with alcohol, carbon disulphide, and ether, then dried at 100° C., when exposed in a porcelain boat, placed in a combustion tube, was almost completely expelled from the retaining vessel at the ordinary temperature. The last traces were driven out at a temperature little above 150° C. Brown vanadium sulphide, in a perfectly dry condition, treated in the same manner, was not altered. It only remained then to prepare mixtures of known amounts of the two sulphides and subject them to the action of the acid vapor. To this end the following experiments were made:

I. 0.1303 gram of vanadium sulphide,
0.1302 gram of arsenic sulphide.

The arsenic sulphide was volatilized without difficulty and left 0.1297 gram of vanadium sulphide.

[1] Bestimmung von Vanadinsäure: Dissertation, Rostock, 1894.
[2] *J. Am. Chem. Soc.*, 16, 578.
[3] *Ibid.*, 17, 682.

II. 0.1290 gram of vanadium sulphide,
0.2242 gram of arsenic sulphide,

gave after exposure of one hour to hydrochloric acid vapor a residue of vanadium sulphide, weighing 0.1297 gram.

III. 0.0828 gram of vanadium sulphide,
0.0582 gram of arsenic sulphide,

left 0.0827 gram of vanadium sulphide.

IV. 0.1306 gram of vanadium sulphide,
0.2028 gram of arsenic sulphide,

gave a residue of 0.1308 gram of vanadium sulphide.

V. 0.1403 gram of vanadium sulphide,
0.2409 gram of arsenic sulphide,

left 0.1404 gram of vanadium sulphide.

The temperature in these experiments was not allowed to exceed 250° C., as beyond that point there is danger of affecting the vanadium and causing its partial volatilization.

The method worked so well and with such evidently favorable results that the following course was adopted in the analysis of a specimen of the mineral vanadinite. 0.2500 gram of air-dried and finely divided material was placed in a porcelain boat; the latter was then introduced into a combustion tube and gently heated in a current of dry hydrochloric acid gas. By this treatment vanadic and arsenic oxides were expelled, leaving lead phosphate and chloride. The receiver contalning the vanadium and arsenic was made alkaline and digested with ammonium sulphide. From the solution of the sulpho-salts the vanadium and arsenic sulphides were set free by a dilute acid. After washing and careful drying these sulphides were separated as indicated in the preceding lines, then changed to oxides and determined in the usual manner. The sum of the total constituents determined as lead oxide, phosphoric oxide, vanadic and arsenic oxides, with some lead chloride, amounted to 0.2501 gram.

The method in addition to being satisfactory in the analytical way, certainly forms a very excellent means of purifying and freeing vanadium from arsenic.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, NO. 17.]

THE SEPARATION OF MANGANESE FROM TUNGSTIC ACID.

BY WALTER T. TAGGART AND EDGAR F. SMITH.

Received October 2, 1896.

THE necessity of obtaining pure tungstic acid from time to time, using wolframite as the starting out material, has frequently suggested the inquiry as to what course would probably prove the best in the quantitative separation of this acid from oxides, such as those of iron and manganese.

In the experiments recorded in this communication only the results obtained from a study of mixtures of a manganous salt and a soluble alkali tungstate will be given. The directions taken in the experimentation were, 1st, to effect the separation by the use of yellow ammonium sulphide in the presence of ammonium chloride; 2nd, to eliminate the acid oxide by the use of an alkaline carbonate.

Following the first course, mixtures of definite amounts of ammonium tungstate and manganous chloride were made. To these was added water and a considerable excess of yellow ammonium sulphide, together with ammonium chloride. The mixtures were digested on a water-bath at 70° C., for several hours, and the vessels containing them were then closed and allowed to stand during the night. The manganese sulphide was filtered out, and, after solution, was changed into sulphate and weighed as such, or it was finally obtained as protosesquioxide in the customary way.

RESULTS.

Manganous oxide present.	Manganous oxide found.
Gram.	Gram.
0.1950	0.2121
0.1949	0.2255
0.1290	0.1708
0.1287	0.1720
0.1291	0.1760

In every trial tungstic acid adhered to the metallic oxide.

In trying the second suggestion the soluble tungstate and the

soluble manganous salt were digested for some hours in a platinum dish, upon a water-bath, with an excess of a ten per cent. potassium carbonate solution, after which the whole was evaporated to dryness, the residue boiled up with water, the manganous carbonate filtered out, washed, and finally converted into protosesquioxide.

RESULTS.

Manganous oxide present.	Manganous oxide found.
Gram.	Gram.
0.1949	0.1516
0.1949	0.1534

Several trial were made using a fifty per cent. solution of potassium carbonate.

RESULTS.

Manganous oxide present.	Manganous oxide found.
Gram.	Gram.
0.1951	0.1745
0.1950	0.1528

The experimental evidence given in the preceding paragraphs leaves no doubt as to the insufficiency of the two methods, which were tried, in effecting the desired separation. It is probable that fusion with an alkaline carbonate will alone answer for this purpose. How complete that course would be can only be ascertained by careful experimentation.

In the course of analysis molybdenum is quite often obtained as sulphide. Its conversion into a weighable form is attended with more or less difficulty. Trials made in connection with its estimation show that if the sulphide, as generally obtained, be dried, then intimately mixed with anhydrous oxalic acid, its careful ignition to trioxide can be made quite rapidly.

RESULTS.

Molybdenum trioxide taken.	Molybdenum trioxide found.
Gram.	Gram.
0.3000	0.3009
0.3000	0.2990
0.1007	0.1011

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, No. 18.]

THE SEPARATION OF BISMUTH FROM LEAD.

BY ARTHUR L. BENKERT AND EDGAR F. SMITH.

Received October 2, 1896.

MANY methods have been suggested to effect this separation. In a recent issue of the *Zeitschrift für angewandte Chemie* (1895, p. 530), Olav Steen reviews thirteen of these methods and concludes that an early proposal of Rose,[1] in which the lead is thrown out as chloride and weighed as sulphate, another by Löwe,[2] in which the bismuth is removed as basic nitrate, and a late suggestion made by Jannasch,[3] *viz.*, the expulsion of the bismuth as bromide from a mixture of lead and bismuth sulphides by an air current carrying bromine are the most satisfactory. At least these methods gave Steen the best results. The separation of bismuth from lead frequently confronts the analyst, and any novelty in this direction cannot be absolutely devoid of interest, hence the present communication, which brings data that may perhaps prove of service in the hands of others who are interested in the solution of this analytical problem.

It will be recalled that Herzog[4] proposed to separate bismuth from lead by precipitating the former as basic acetate. The method required considerable time for execution, and in other hands than those of its author apparently has not yielded entirely satisfactory results.

An idea closely related to that of Herzog would be the substitution of a formate solution for that of the acetate. This was done with results that are very interesting.

Solutions of lead nitrate and bismuth nitrate in nitric acid were made up of such strength that twenty cc. of the first contained 0.2076 gram of lead oxide, and twenty cc. of the second 0.1800 gram of bismuth trioxide. The lead and bismuth were accurately determined after dilution to a liter. Twenty cc. of these two nitrate solutions were then introduced into a beaker

[1] *Ann. chem. phys. Pogg.*, 110, 425.
[2] *J. prakt. Chem.*, 74, 348.
[3] *Ber. d. chem. Ges.*, 25, 124.
[4] *Ztschr. anal. Chem.*, 27, 650.

glass, carefully diluted and almost neutralized with sodium carbonate, or until the incipient precipitate dissolved slowly, when considerable sodium formate solution of sp. gr. 1.084 and a few drops of aqueous formic acid were added. The total dilution of the liquid was 250 cc. It was gradually heated to boiling and held at that point for *five* minutes. The precipitate was then allowed to subside, but was filtered while yet hot. The basic formate separates rapidly and is easily washed if not boiled too long. It was washed with hot water, then dissolved in dilute nitric acid and precipitated with ammonium carbonate. The ignited bismuth trioxide weighed too much; it contained lead. However, the impure oxide was dissolved in nitric acid, diluted to 250 cc., and after the addition of sodium carbonate to almost complete neutralization, sodium formate and free formic acid were added as before, and *the precipitation of basic formate repeated*. This precipitate after solution and the bismuth thrown out by ammonium carbonate gave 0.1804 gram of bismuth oxide instead of 0.1800 gram as required by theory. Seven additional separations, in which the quantities of bismuth and lead were the same as indicated above, gave:

0.1806 gram of Bi_2O_3.
0.1806 " " "
0.1803 " " "
0.1804 " " "
0.1804 " " "
0.1805 " " "
0.1796 " " "

The conditions in these determinations were similar to those previously outlined.

With a solution containing 0.3600 gram of bismuth oxide and 0.2076 gram of lead oxide, operating in an analogous manner, two results were obtained:

0.3595 gram of Bi_2O_3.
0.3605 " " "

instead of the required 0.3600 gram.

The residual bismuth trioxide was examined for lead, but none was found.

[CONTRIBUTIONS FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF CINCINNATI.]

XLVIII.—ON SOME NEW FORMS OF GAS GENERATORS.[1]

BY THOMAS H. NORTON.

Received August 27, 1896.

IMPROVEMENTS in the construction of the automatic generators, for the gases most frequently used in our laboratories, are always welcome. The following three types, which I devised some time since, have been subjected to prolonged trial in the laboratory of the University, and have given such satisfactory results, that a detailed description would seem worthy of publication.

In Fig. 1 is represented a gas generator for hydrogen, hydrogen sulphide, etc., which differs in several details from well known types of the same general outline. It is constructed of glazed earthenware, and is easily made in our ordinary potteries. *A*, the outside container, is provided with handles on the outside, and is ordinarily sixty cm. in height. Its chief peculiarity is the presence on opposite sides of the inner wall, of the shoulders *DD*, each about four cm. wide and slightly concave on the lower surface. *B*, the gas reservoir, is of the ordinary bell-jar construction, with orifice at the top for the introduction of a perforated stopper and outlet tube. It is provided with projecting shoulders three cm. wide, corresponding to *DD*, and at such a height that they barely slip beneath the latter. At the bottom are frequent circular perforations, one centimeter in diameter, to allow of the easy passage of the acid charge. The recipient *C*, designed to hold zinc or any solid charge, is provided with a loose disk perforated with many fine openings and resting upon the shoulder of the constriction. Beneath the constriction are perforations corresponding to those in *B*. A strong copper wire or rod, passing through the perforations of both parts of the apparatus, holds *B* and *C* in their mutual position to each other.

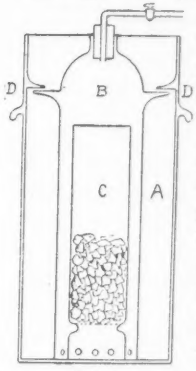


FIG. 1.

[1] Read before the American Association for the Advancement of Science at the Buffalo Meeting.

The working of the generator is exceedingly simple. *C* receives its charge of zinc, marble or ferrous sulphide. *B* is placed over it. The copper rod is passed through the perforations at the bottom. *B* with *C* is then introduced into *A*, and turned until the shoulders of *B* are beneath *DD*. *A* is then filled with the acid charge. The buoyancy of *B* is partly overcome by the rigid attachment of *C*, and entirely prevented by *DD*. Gas can be drawn off as desired, by opening the tap at the outlet tube. When, as naturally occurs, the acid in the lower portion of the generator becomes weak and the evolution of gas sluggish, the exit tap is closed, *B* is turned slightly so as to be free from *DD*, and is then lifted, by grasping the neck, along with the holder *C*, until entirely above the surface of the acid. Both are then plunged to the bottom of *A*, and a few repetitions of this churning movement renders the acid charge of uniform strength.

This style of generator has rendered excellent service. For example, one sixty centimeters in height easily supplies all the hydrogen sulphide required by a class of thirty in qualitative analysis. The special advantages of this generator are to be found in the ease and simplicity with which the buoyancy of the gas reservoir is overcome and the acid charge is maintained at a uniform strength until practically exhausted.

In Fig. 2 we have a less compact and less transportable form, but one which maintains the uniform strength of the acid charge until it is exhausted, without the need of special manipulation, as described above. It is particularly designed for use where small amounts of hydrogen sulphide are in constant requisition, as in the laboratory for qualitative analysis, and it has the advantage of being capable of easy construction from the glassware found in any well equipped laboratory. *A* is a capacious tubulated bell-jar inverted and resting upon

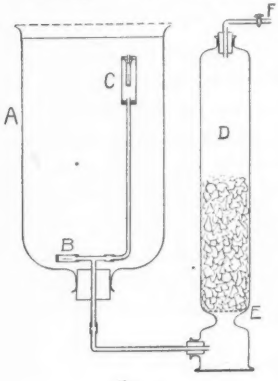


FIG. 2.

either a tripod or the ring of an ordinary support. The perforated stopper in the neck is traversed by a ⊤ tube. One terminal of this tube is connected with a simple Bunsen valve, *B*, *i. e.*, a piece of rubber tubing, closed at one end and provided with a clean cut slit in the rubber some two cm. in length. The other terminal of the ⊤ tube is connected with *C* in the upper portion of *A*. The attachment *C* is similar to that frequently introduced between suction pumps and filtering flasks. It is the reverse of *B* in its construction, allowing a current of liquid to enter from the outside through the rubber valve. *A* serves as a reservoir for the acid charge. The third external terminal of the ⊤ tube is connected with the tubulus of the lower portion of an ordinary lime drying tower, *D*, preferably of the largest size constructed. *D* serves as the recipient for the ferrous sulphide, etc., which may be used, and is provided with a perforated disk at *E* and the outlet tube *F*, the latter on a level with the top of *A*. The working of the generator is exceedingly simple. *A* is charged with acid and *D* with, say, ferrous sulphide. When *F* is opened the acid flows through *C* into *D*. When *F* is closed the pressure of the gas evolved forces the acid back into *A* through *B*. The result is that the supply of acid furnished *D* is always from the top of the reservoir *A*, and hence stronger than that found in the lower strata, which are successively of greater specific gravity, weaker in acid and richer in saline matter, as the bottom is approached. The arrangement permits of a very complete utilization of the acid. When the current of gas is in continuous demand, and evolution becomes sluggish, it is necessary to close the tap at *F* for a short time until the liquid in *D* is driven back into *A*.

Care must be exercised in constructing the valve at *C*, so that it will yield to a very slight pressure. To effect this the slit in the rubber should be at least two cm. in length. When the apparatus is used exclusively for the evolution of hydrogen sulphide to be employed in qualitative analysis, it is desirable to have beyond *F* some device which regulates uniformly the strength of the current of gas and keeps it within the limits of easy absorption. In practice this has been accomplished most simply by introducing into the rubber tube attached to *F* a short

piece of glass tubing, one end of which is drawn out so as to form a very narrow opening.

Essentially the same principle for the control of the strength of the acid charge is to be found in the generator devised recently by Professor Harris. In consequence of the costly character of the latter, due largely to the use of valves of elaborate construction, the form of generator just described may be welcome to many on account of its simplicity and inexpensiveness.

An automatic chlorine generator based upon the use of manganese dioxide, has long been desired. In Fig. 3 is shown such a generator which for six years has rendered satisfactory service, both on the lecture table and in the laboratory. The essential parts only are outlined without the accompanying supports. *A* is a copper funnel, provided with a hollow projection *C*, on one side, perfectly similar in make to the funnels used for hot water filtration. It can be advantageously replaced by the more graceful and modern type of aluminum funnel, resting in a ring burner. The reservoir *B* is of glass, and is an article of current manufacture, obtainable from all dealers in chemical glassware. The long, tapering neck is tightly fastened in the neck of the funnel by means of a section of rubber tubing. A large opening at the top, closed by a rubber stopper, serves for the admission of the charge. In a smaller tubulure on the side is a perforated rubber stopper with outlet tube and tap. The funnel with its reservoir is held firmly in a support, so that the end of *C* is about two cm. above the top of an ordinary burner. A perforated plate is introduced into *B* so as to prevent solid matter from falling into the narrow neck. The latter is connected at *D* with a large tubulated bottle *E*, which serves as a reservoir for hydrochloric acid, and is attached to a support so that it can be raised

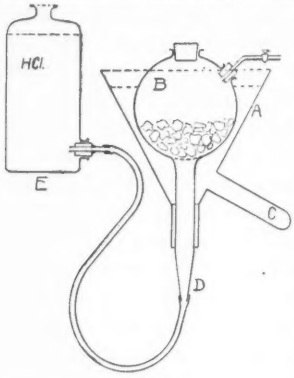


FIG. 3.

or lowered at will. When in use *B* is filled to two-thirds of its capacity with manganese dioxide, large lumps alone being used, as powdered mineral may easily cause a stoppage of the connections. *E* is filled with hydrochloric acid and raised to a level slightly above the top of *B*. Water is poured into the funnel *A* until it is nearly full, and a lamp is placed under *C*. As soon as the temperature has reached about 80°, a very small flame suffices to maintain the activity of the generator. When the exit from *B* is open, the acid enters and the evolution of chlorine continues until checked by closing the tap, when the acid is driven back into *E*. A slight agitation of the latter before opening the tap serves to prevent the accumulation of a stratum of weak acid at the bottom. It is advisable to lower the reservoir *E* when a current is not required, so as to avoid pressure and any possible escape through minute leaks. In practice it is also found desirable to connect the opening of *E* by a flexible tube with a bottle of caustic soda solution, the tube terminating at the surface of the solution. This prevents any escape into the surrounding air of chlorine, with which the contents of *E* are soon saturated. When thus arranged a current of the gas can be taken at will from the generator, the sole condition being the maintenance of a small flame beneath *C*. The manifold advantages of such a device, especially for the lecture table, will be appreciated by all who attempt an extended series of experiments with chlorine. As described above the generator can be readily constructed from pieces of apparatus ordinarily found in a well equipped laboratory. I have found a generator in which the reservoir *B* contains 1500 cc., a very convenient size for use in the lecture room.

MINERAL CONSTITUENTS OF THE WATERMELON.

By George F. Payne.

Received September 28, 1896.

THE watermelon is not a crop that is widely grown even in this country with great success. It is this very reason which makes it a desirable crop to handle in Georgia, as the watermelons in this state attain finer flavor, crispness, juiciness and sweetness than anywhere else in the world.

Upon analysis of two medium-sized watermelons cut up and mixed together, we found them to contain just one-third per cent. of pure ash, calculated as free from carbonic acid. The exact figures were 0.3338, which in our calculations we will round off into an even one-third, which it practically is.

The composition of watermelon ash is as follows:

	Per cent.
Sulphur trioxide	4.41
Calcium oxide	5.54
Magnesium oxide	6.74
Potassium oxide	61.18
Sodium oxide	4.31
Silicon dioxide	2.15
Phosphorus pentoxide	10.25
Chlorine	4.94
Iron sesquioxide	0.48
Total	100.00

A good average crop of watermelons is considered to be about one-half carload to the acre, though much larger crops than this are sometimes made. Large watermelons are also considered desirable, hence in considering what is carried off from the land by the removal of the crop, it is well to consider how much would be taken off by a large crop, as it is the large crops which we desire to produce. We have before us a report of a crop of watermelons upon an acre of land which is an unusually large one, but which was weighed up in the presence of disinterested witnesses and sworn to by them as being honestly grown upon an acre and correctly weighed. This crop weighed 39,766 pounds. One-third per cent. of such a crop would be pure ash, and consequently the mineral plant food taken out of an acre of land by such a crop would be as follows:

	Pounds.
Sulphur trioxide	5.85
Calcium oxide	7.34
Magnesium oxide	8.93
Potassium oxide	81.09
Sodium oxide	5.71
Silicon dioxide	2.85
Phosphorus pentoxide	13.59
Chlorine	6.55
Iron sesquioxide	0.64
Total	132.55

In the crop mentioned above to replace the phosphoric acid and potash carried off from one acre by the melons alone, not taking into account the vines and roots, would require:

	Pounds.
Acid phosphate (thirteen per cent. P_2O_5)	100
Muriate of potash (fifty per cent. K_2O)	160

A fair crop of melons upon good land, however, is usually considered to be about one-third of the above large crop or about one-half carload. If we estimate then the amounts of phosphoric acid and potash required for an average crop of fair character, such a crop will take from the soil materials to replace which will require about:

	Pounds.
Acid phosphate	33⅓
Muriate of potash	53⅓

This will give about four and one-half pounds of available phosphoric acid to an acre, and about twenty-seven pounds of pure potash to an acre. The usual goods on the market guarantee about ten per cent. of available phosphoric acid and about one per cent. of potash. The use of 300 pounds of such goods upon each acre of watermelons, furnishes thirty pounds of available phosphoric acid, or about six and one-half times as much as is needed to replace what is carried off by the watermelons. It also furnishes about three pounds of potash, which is only one-ninth of what is carried off by the crop removed. This being the case it shows with what advantage and economy the watermelon grower can replace a large proportion of his phosphoric acid with potash.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE U. S. DEPARTMENT OF AGRICULTURE, NO. 22.]

A MODIFIED FORM OF THE EBULLIOSCOPE.

BY H. W. WILEY.

Received September 26, 1896.

THE determination of the alcohol in wines and beers, from the temperature of the vapors given off on boiling at atmospheric pressures, has long been practiced. The instrument by means of which this determination is made is known as the ebullioscope or ebulliometer. The use of this instrument

was proposed many years ago by Tabarié, and it has been improved by Malligand, Salleron and others.

It is evident that if so simple an apparatus could be made to give accurate data, it would come into general use for ordinary purposes. The difficulties which have attended the use of the ebullioscope, however, have been of such a nature as to render the data given by it somewhat unreliable. Among these difficulties may be mentioned the fact that a wine or beer contains a considerable quantity of dissolved matters, which serve to render the temperature of the boiling liquid higher than the temperature of a mixture of a similar percentage of alcohol with water. While the temperature of the vapors emitted are, theoretically, not influenced in a marked degree by the initial temperature at which they are formed, nevertheless, in practice it has been shown that the tendency of the higher initial boiling point is to give a higher reading to the thermometer whose bulb is surrounded by the emitted vapors.

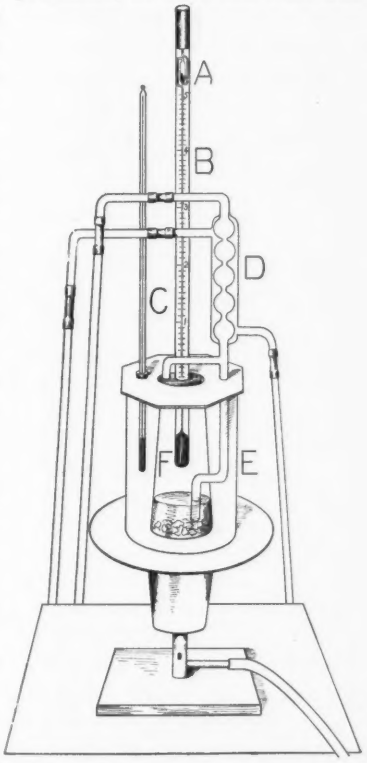
Another difficulty attending the use of the ebullioscope is found in the fact that the percentage of alcohol in the vapors emitted is much greater than in the residual liquid. As a result, it is difficult to establish a balance between the condensed vapors and the liquid remaining in the flask, in such a manner as to secure a continuous evolution of a vapor containing a definite proportion of alcohol.

In the third place, it has been customary to return the condensed vapors through the apparatus in such a way that they come in contact with the uncondensed vapors surrounding the thermometer. By this means the vapors surrounding the bulb of the thermometer are subjected to changes of temperature which render it difficult to get a mean reading of the height of the mercurial column in the instrument. The variations which the mercurial column may undergo amount, in some instances, to two or three-tenths of a degree and as each tenth of a degree represents approximately a tenth of a per cent. of alcohol, it is not difficult to see that these variations would tend to lead to erroneous results.

In the fourth place, barometric changes, which are constantly taking place in the atmosphere, change the boiling point of the

vapor of water so that it is frequently necessary to check the instrument with pure water, in order to have an initial temperature for the calculations.

In the apparatus which is presented, an effort has been made to remedy the difficulties which have been mentioned above. The apparatus consists of the flask *F*, which is closed by a rubber stopper carrying the large thermometer *B* and a tube leading to the condenser *D*. The vapors which are given off during ebullition are condensed in *D* and return to the flask through the tube, as indicated in the figure, entering the flask below the surface of the liquid therein.



The flask is heated by a gas lamp and is placed upon a circular disk of asbestos in such a way as to entirely cover the hole in the center of the asbestos disk, which is a little smaller than the bottom of the flask. The whole apparatus is protected from external influences of temperature by the glass cylinder *E*, which rests upon the asbestos disk below and is covered with a detachable, stiff rubber cloth disk above.

The thermometer *C* indicates the temperature of the ambient air between *F* and *E*. The reading of the thermometer *B* should always be made at a given temperature of the ambient air, as indicated by *C*. The tube leading from the top of the conden-

ser *D* to the left, is made long and is left open at its lower extremity, in order to secure atmospheric pressure in *F*, and at the same time prevent the diffusion of the alcohol vapors through *D*.

The flame of the lamp is so regulated as to bring the temperature of the thermometer *C* to about 90° in ten minutes for substances not containing over five per cent. of alcohol. After boiling for a few minutes, the temperature, as indicated in the thermometer *B*, is constant, and the readings of the thermometer should be made at intervals of about half a minute for two minutes. Some pieces of scrap platinum placed in the flask will prevent bumping and secure a more uniform evolution of vapor.

Slight variations, due to the changes in temperature of the vapor, are thus reduced to a minimum effect upon the final results.

The apparatus is easily operated, is quickly charged and discharged and with it at least three determinations of alcohol can be made in an hour.

The thermometer used is the same as is employed for the determination of freezing and boiling points in the ascertainment of molecular weights. The reading of the thermometer is arbitrary, but the degrees indicated are centigrade. The thermometer is set in the first place by putting the bulb in water containing sixteen grams of common salt to 100 cc. When the water is fully boiling, the excess of mercury is removed from the column in the receptacle at the top and then, on placing in ordinary boiling water, the column of mercury will be found a little above the 5° mark. This will allow a variation in all of 5° in the temperature, and a thermometer thus set can be used for the estimation of percentages of alcohol from one to five and a half, by volume. When the liquor contains a larger percentage of alcohol than this, it is advisable to dilute it until it reaches the standard mentioned.

In order to avoid frequent checking of the thermometer, rendered necessary by changes in barometric pressure, I use a second apparatus made exactly as the one described, in which

water is kept constantly boiling. It is only necessary in this case to read the two thermometers at the same instant in order to make any necessary correction required by changes in barometric pressure.

It is not my purpose here to submit a table showing the percentages of alcohol corresponding to any given depression in the temperature of the boiling vapor. It is only necessary to call attention to the fact that for the percentages named, the platted line showing the variation in depression from 0° to five per cent. by volume of alcohol is practically straight and that for each 0.8° change in the boiling point of the vapor, there is a change of about one per cent by volume of alcohol. This rule can be safely applied for practical purposes to all liquors containing not more than five and five-tenths per cent. of alcohol. For instance, if, in a given case, the temperature of the vapor of boiling water, as marked by the thermometer, is 5.155°, and the temperature of the vapor of a sample of beer is 2.345°, the depression is equivalent to 2.810°, and the percentage of alcohol by volume is therefore 2.81 divided by 0.80 = 3.51.

The thermometer used is graduated to hundredths of a degree and is read by means of a cathetometer, which will easily give readings to five thousandths of a degree.

The reading of the thermometer is facilitated by covering the bulb with a test-tube containing water. The high specific heat of the water distributes evenly any little variations of temperature which otherwise would cause the mercurial column in thermometer *B* to oscillate. The water jacket also serves as a protection against the projection of any particles of the boiling liquor directly against the bulb of the thermometer.

It is believed that this apparatus is the best form of ebullioscope which has yet been offered for practical use to analysts.

VOLUMETRIC DETERMINATION OF ACETONE.[1]

BY EDWARD R. SQUIBB.

Received November 9, 1896.

IN the *Moniteur Scientifique* of 1893, 41, 4 Serie, Vol. 7, 1°, p. 272–274, MM. J. Robineau and G. Rollin publish a paper entitled, "Dosage Volumetrique de L'Acetone," and the following is, first, a free translation and abridgement of this paper; and second, the detail of an improvement of the process whereby it is rendered easier, more simple, quicker, and better adapted to technical uses, whilst still sufficiently accurate for most purposes.

FIRST, FREE TRANSLATION.

The common way of determining the proportion of acetone in a liquid containing it is to convert the acetone into iodoform by means of iodine in the presence of soda after eliminating from the liquid everything that would interfere with the proper reaction.

For this process binormal solutions of iodine and of sodium hydroxide are used, and the precipitated iodoform is washed, dried, and weighed, or is dissolved in ether, and the whole or a fraction of the ethereal solution is dried over sulphuric acid and weighed.

The appreciable volatility of iodoform at ordinary temperatures introduces a source of error that is objectionable, especially when dealing with small quantities.

But, aside from this, the time required for this process is relatively so long that we have sought to change it to a volumetric process that is more rapid.

Our proceeding consists in mixing the acetone with a solution of potassium iodide and sodium hydroxide, and then transforming it into iodoform with a titrated solution of a hypochlorite. The end reaction is indicated by the appearance of a blue color, when a drop of the liquid is touched with a drop of bicarbonated starch solution.

From the quantity of hypochlorite used the quantity of acetone is deduced.

[1] Read before the New York Section of the American Chemical Society, November 6th, 1896.

For it happens that the presence of even the smallest trace of an alkaline hypoiodite, in a solution of soda, gives a blue color with a starch solution which contains an excess of sodium bicarbonate.

Again, a liquid containing acetone, an iodide and caustic soda in excess, and into which a solution of hypochlorite is passed, gives no reaction with bicarbonated starch until the whole of the acetone is converted into iodoform.

This proceeding, however, only gives constant results when certain precautions are taken. Unless the liquid containing the acetone be sufficiently alkaline, an excess of hypochlorite will be required to decompose all the acetone.

The potassium iodide must be in excess.

The dilution must be fairly uniform, and the concentration of the hypochlorite about the same for the different titrations.

The process should not be used in too strong a light.

It is very important that the liquid should be constantly stirred during the additions of the hypochlorite.

The strength of the hypochlorite solution is ascertained by trial against a pure acetone made by the bisulphite process.

PREPARATION OF THE HYPOCHLORITE.

For the titration of liquids containing considerable proportions of acetone, the hypochlorite solution is prepared as follows:

To 500 cc. of the concentrated solution of sodium hypochlorite of commerce, which tests from forty-five to fifty-five volumes of chlorine, an equal measure of water, and ten cc. of solution of pure soda of 36° B., are added, and the solution is kept in an amber colored bottle, well corked.

TITRATION OF THE HYPOCHLORITE SOLUTION.

About two grams of pure acetone from bisulphite is weighed off and diluted to 500 cc.

Then ten grams of pure potassium iodide is put into a conical Bohemian beaker and 100 cc. of the diluted acetone and twenty cc. of solution of caustic soda of 28° B. are successively added, and the whole is stirred until the iodide is dissolved and the liquid is homogeneous.

Into this the hypochlorite solution is passed drop by drop from a burette, with constant stirring, precipitating the iodoform in large flakes which easily settle out. When farther additions of the hypochlorite give but a light cloudiness a drop of the liquid is transferred to a white porcelain plate by means of a glass rod and is there brought in contact with a drop of the bicarbonated starch solution. As soon as the hypochlorite is in excess the blue color appears very distinctly. The volume of hypochlorite used is then read off from the burette, and then for security of result the titration is repeated.

Example.—2.081 grams pure acetone is weighed off and diluted to 500 cc; 100 cc. of this solution requires 22.5 cc. of the hypochlorite. This gives for each cc. of hypochlorite 0.01874 gram of pure acetone. These results are liable to vary a little if the conditions of the experiment vary much. The stirring is supposed to be constant, and the hypochlorite solution to be regularly added.

If to 100 cc. of the diluted acetone 100 cc. of water be added and the same quantities of iodide and soda as above, 22.05 cc. of hypochlorite is required instead of 22.5 cc. In using forty cc. of the soda solution instead of twenty cc., twenty-two cc. of the hypochlorite is required. In using sixty cc. of soda solution instead of twenty cc., 21.6 cc. of the hypochlorite is required. In using ten cc. of soda instead of twenty cc., twenty-three cc. of the hypochlorite is required.

These results show that dilution of the acetone and a small excess of soda have but little influence, but that a deficiency in alkalinity has a very considerable effect on the quantity of hypochlorite required. And farther, that the alkalinity indicated by twenty cc. of soda solution of 28° B. appears to be normal.

Under the given conditions of alkalinity and dilution the relations between acetone and the available chlorine of the hypochlorite is obviously one molecule of acetone to six atoms of chlorine.

The solution of hypochlorite used by us was the liquor of Penot, testing 21.56 volumes.

The titration of the hypochlorite with pure acetone may be

omitted, simply determining the available chlorine of the liquor of Penot instead, but we prefer the titration with pure acetone.

DETERMINATION OF ACETONE IN A COMPLEX LIQUID.

We give as an example of this the titration of a complex liquid made with precision, which liquid has served us to control the accuracy of the process.

The complex liquid contained :

1.510 grams	water,	39.98 per cent. acetone.
1.677 "	ethyl alcohol,	
1.550 "	methyl alcohol, pure	
3.149 "	acetone, pure from bisulphite	

Of this mixture 3.2445 grams was weighed off and diluted to 500 cc. Proceeding as before 100 cc. of this dilution, ten grams of potassium iodide, and twenty cc. of soda solution of 28° B. required 13.85 cc. of the hypochlorite. This by calculation gives 39.99 per cent. of acetone, and thus verifies the composition of the complex liquid; and it is seen that the presence of ethyl alcohol is without influence on the result.

The effect of the presence of paraldehyde in the same complex liquid was tried by a similar titration.

To 100 cc. of the complex solution corresponding to 0.4162 gram of pure acetone, ten cc. of an aqueous solution containing five per cent. of pure paraldehyde was added (say one-half gram) or a little more paraldehyde than acetone. This mixture took 22.4 cc. of the hypochlorite instead of 22.2 cc. as required.

This variation is slight for the relatively large proportion of paraldehyde, and is greater for larger proportions, but instances are rare in which paraldehyde is present in such proportions.

In all such instances where the presence of the aldehyde has been established by the process of Bardy, the acetone should be purified by this process before titration.

For the determination of acetone in very dilute solutions a solution of hypochlorite of one-fifth of the above strength is preferred. That is, a solution containing four or five volumes of available chlorine, and the degree of alkalinity should be proportionately reduced.

With a little practice it is easy to judge as to how much acetone is present in a liquid to be titrated, and from this to judge

of the corresponding quantity of hypochlorite required, and in this way keep the conditions of the method nearly uniform, and the more uniform the conditions the more constant the results.

This process has the great advantage of being rapid, and thus of permitting a number of titrations being made in a short time with results sufficiently accurate.

REMARKS.

The reaction used in this titration is very delicate, and where traces of acetone are concerned it is better seen when there is excess of iodide and of soda and but little hypochlorite. An aqueous solution of 0.004 gram of acetone in the liter gives a heavy cloudiness immediately. The reaction with 0.0012 gram of acetone in the liter is seen in a few moments. With 0.0008 gram to the liter the reaction is difficult to see. This reaction should not be made in a bright light. In sunshine or in a very bright light the traces of iodoform produced disappear very rapidly, the liquid becoming clear, but in a dim light the precipitate does not disappear.

The titrated solution of hypochlorite should be kept in amber-colored glass in a cool place and sheltered from bright light. The titration should be frequently repeated, because it varies rather rapidly, especially when diluted. We have made a series of experiments on this point, which strikingly show these variations under different influences. A solution of hypochlorite prepared for titrations gave 22.16 volumes of available chlorine; kept in a cool place, in obscurity for six days, it gave 21.96; kept in colorless glass, corked, in a bright light, most of the time in sunlight, for seven days, it gave 12.32. In a water-bath at 100° C. for a quarter of an hour it gave, when cooled, 19.48.

END OF TRANSLATION.

The rapidly increasing uses of acetone in the three years that have passed since the publication of this important paper of Robineau and Rollin have given to it so much additional importance that it seemed well to the present writer—who early adopted this volumetric method—to attempt to modify the method in the direction of greater simplicity and rapidity, even

if this should be at the cost of a little of its accuracy. As acetone comes more and more to take the place of both ethyl and methyl alcohol as much the better solvent for most purposes, and as its manufacture is cheapened, it becomes more and more desirable to have a rapid and easy way of estimating its proportions in mixtures or under conditions to which specific gravity is not applicable.

Therefore, taking the above quoted paper as a basis, and giving full credit to the authors of it for every important principle and step of the method, the following slight modifications are offered as the result of about three months' experience with the original process and over a year's experience with the modifications.

STANDARD SOLUTION OF ACETONE.

A flask of 100 cc. capacity containing about fifty cc. of distilled water is carefully weighed. To this is added about thirteen cc. of pure acetone, made by the bisulphite process. The weight is then again taken, when it will be found that the acetone added is a fraction more or less than ten grams. The dilution is then transferred to a measuring flask, the weighing flask being rinsed in and is farther diluted with distilled water until each ten cc. of the dilution contains one-tenth gram of acetone. This is kept in a well stoppered bottle of dark glass, for, although the writer has no evidence of any change taking place in acetone, and believes it to be quite as permanent as ethyl alcohol, still it may be well to keep a dilute standard solution protected against bright light.

Of this solution or dilution ten cc. equal to one-tenth gram of acetone, is accurately measured off for each titration of the solution of hypochlorite.

SOLUTION OF POTASSIUM IODIDE.

Of this salt 250 grams are dissolved in distilled water, and the solution is made up to one liter, when each ten cc. will contain two and a half grams of the iodide.

SOLUTION OF SODIUM HYDROXIDE.

Of commercial caustic soda, purified by alcohol, 257 grams is dissolved in distilled water, the solution made up to one liter,

and set aside until it settles quite clear. Then 850 cc. of clear solution is poured off and added to the solution of potassium iodide, making 1,850 cc. of total solution.

Of this solution twenty cc. is taken for each titration.

The remainder of the soda solution is again allowed to settle clear for farther use in the hypochlorite solution.

SOLUTION OF SODIUM HYPOCHLORITE.

The officinal solution of chlorinated soda of the U. S. Pharmacopœia ("Liquor Sodæ Chloratæ," U. S. P.) answers very well for this process, the officinal strength of two and six-tenths per cent. of available chlorine being quite convenient.

To a liter of this solution in a bottle of dark glass, twenty-five cc. of the above described clear soda solution is added and the mixture well shaken.

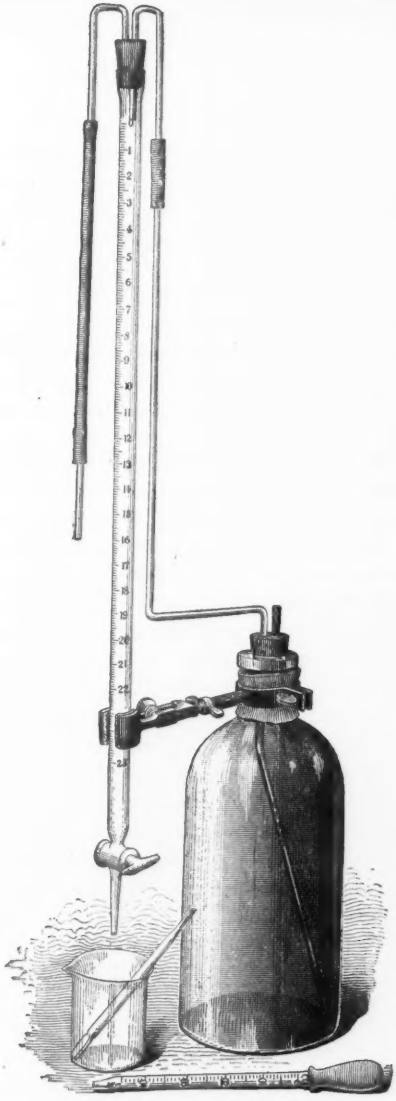
If in buying the "Solution of Chlorinated Soda" of the U. S. P. for this process it should be found, as is not unfrequently the case, weaker than is required by the U. S. P., or, if by keeping it becomes weaker, this will be at once discovered on balancing it against the standard acetone solution, and so long as the one-tenth gram of acetone does not require more than say twenty cc. of the more dilute hypochlorite, the formula need not be modified.

If there be much of this titration to do it is very convenient to fit this bottle with an automatic zero burette,[1] as shown in the following illustration, this form being, so far as is known, original with the writer and very convenient for general rapid working with a burette. The advantage is, beside that of rapid and easy working, that it does not require a special burette and is easily fitted up from the resources of any laboratory.

BICARBONATED STARCH SOLUTION.

Starch, 0.125 gram, is mixed with five cc. of cold water, and then added to twenty cc. of boiling water and boiled. When cold two grams of sodium acid carbonate is added and stirred until dissolved. Kept in a colorless bottle this solution does not sensibly diminish in delicacy or reaction in three months. But for how much longer it would remain good for this reaction was not tried.

[1] This Journal, 16, 145.



AUTOMATIC ZERO BURETTE.

THE TITRATION.

The burette being filled with the solution of sodium hypochlorite, ten cc. of the standard solution of acetone (equal to one-tenth gram of acetone) is measured into a beaker of about fifty cc. capacity, and twenty cc. of the mixed solution of iodide and soda is added and stirred well. Into this the hypochlorite solution is passed in rapid dropping, with constant stirring, until eight or ten cc. has been run in. Then the precipitated iodoform is allowed to settle out, and a drop or two more hypochlorite is added. Should this produce a dense cloudiness one-half cc. more hypochlorite is added, and well stirred and again allowed to settle. Then a drop or two more of hypochlorite is added. If there should still be a cloudiness, another one-half cc. of the hypochlorite is added and well stirred, and so on until the cloudiness is very slight. Then the starch testing begins.

A small drop of the liquid is transferred by a rod to a white porcelain tile or plate, and a similar small drop of the starch solution is placed very near it. Then with the first rod the drops are made to connect by a fine line, so that the whole has a dumb-bell form. If there be no blue color, one or two-tenths cc. more of the hypochlorite is added and well stirred, and the testing is repeated, until finally a blue line will be seen at the moment of contact of one drop with the other. If the last negative testing has taken 10.4 cc. from the burette, and this positive testing, which has given the blue line, required 10.6 cc., then the accepted reading would be 10.5 cc., and this would be the hypochlorite equivalent of one-tenth gram of acetone. If the blue line be very faint, it will be momentary only, and will indicate that the excess of hypochlorite is very small, and that 10.6 cc. is a closer reading than 10.5, but the process is not sufficiently accurate to take much account of such differences, since even with much experience and great care it is hardly practicable to get any two titrations to agree within one-tenth cc. of hypochlorite.

Having then 10.5 cc. as the hypochlorite equivalent of one-tenth gram of acetone at this time, it is easy to estimate any smaller or larger quantity of acetone that requires a smaller or

larger quantity of the hypochlorite by the equation 10.5 : 0.1 : : $a : x$.

But this hypochlorite solution is liable to diminish in strength by keeping, and therefore must be standardized by this standard acetone solution as often as the accuracy of the determinations may require. At times the change in strength is scarcely perceptible from day to day in several successive day's work, but in standing for a week or two there will always be a falling off in strength to the extent of one-tenth to five-tenths cc. in the hypochlorite. The addition of the soda solution appears to render the hypochlorite more permanent, just as the sodium bicarbonate renders the starch solution more permanent. But in the case of the starch the blue reaction does not occur if the bicarbonate be not present.

The titration of the acetone present in unknown dilutions requires first that the strength should be estimated by known conditions or by sensible properties, in order to keep the proportions of the reagents and the dilutions approximately the same, or at least not differing very widely when close determinations are required. If then the taste and smell should indicate that the acetone to be tested is below twenty-five per cent., four-tenths cc. may be taken for the testing. If over twenty-five per cent. and under fifty per cent., two-tenths cc. may be taken. If over fifty per cent., one-tenth cc. is sufficient.

For the adjustment of these small quantities with a sufficient degree of accuracy for rapid technical working, it is convenient to have a five-tenths cc. pipette divided in 0.01 cc. fitted with a rubber bulb, as shown in the illustration. By screwing the neck of this bulb up or down upon the glass, with the point in the liquid, close measurements may be quickly made.

A beaker of fifty cc. capacity containing ten cc. of water is weighed and the weight noted. The four-tenths, two-tenths, or one-tenth cc. of the sample to be titrated is delivered in the water and the weight again taken to give the quantity of the sample taken for the titration. Then the twenty cc. of the iodide and soda solution is added, the whole well stirred, and the hypochlorite dropped in, and the end reaction managed precisely as described in standardizing the hypochlorite, and the quantity of

hypochlorite used is noted. Then as 10.5 cc. of the hypochlorite is to one-tenth gram of acetone, so is the quantity of hypochlorite now used to the quantity of acetone present in the portion of the sample taken for titration. Then as the weight of this portion taken for titration is to the quantity of acetone found in it, so is 100 to the percentage of acetone in the sample.

For example, a sample supposed to be not far from absolute is to be titrated. A fifty cc. beaker with ten cc. of water weighs 25.283 grams; with one-tenth cc. of the sample added the weight becomes 25.360 grams, giving 0.077 gram as the weight taken for the titration. To this is added the twenty cc. of iodide and soda solution, and the mixture being well stirred, the hypochlorite is dropped into saturation when seven and nine-tenths cc. is found to have been used. Then as 10.5 is to one-tenth, so is seven and nine-tenths to 0.0752 gram of acetone in the 0.077 gram of the sample taken. Then as 0.077 gram of the sample taken is to the 0.0752 of acetone indicated, so is 100 to 97.66 per cent. of acetone in the sample.

This is the rationale of the operation, but the calculation is shortened by simply dividing the standard hypochlorite (10.5 cc.) into the hypochlorite required (seven and nine-tenths cc.) to get the corresponding acetone (0.0752 gram), and then dividing the weight of the sample taken (0.077 gram) into the weight of acetone obtained from it (0.0752 gram) to get the percentage proportion of the acetone (97.66 per cent.).

Of course the method of definite dilution, and the titration of an aliquot part, as described in the original paper of Robineau and Rollin (see translation) is available and more accurate than that here recommended, and takes but little more time.

Where acetone is made, or is much used, and especially in processes where it is recovered by distillation to be used over again, there is often much need of testing the strength of very weak dilutions, and of knowing when acetone is absent. In many such uses accuracy is not required and rough estimates are sufficient. For work of this kind, especially when the strength is below ten per cent., the weighing of the sample to be tested may be omitted, because the specific gravity is so nearly

that of water that the measure may be accepted as cubic centimeter for gram.

DETERMINATION OF ACETONE IN THE PRESENCE OF ETHYL ALCOHOL.

The standard dilution of acetone containing ten grams in the liter was used, and ten cc. of this required 14.3 cc. of the hypochlorite solution. On repetition 14.4 cc. was required.

A dilution of ethyl alcohol was made containing ten grams in the liter, and ten cc. of this requires one-tenth cc. of the hypochlorite. On repetition 0.125 cc. was required.

To ten cc. of the acetone dilution two-tenths cc. of the alcohol dilution was added, and this mixture required 14.4 cc. of the hypochlorite solution. On repetition 14.4 cc. again was required.

To ten cc. of the alcohol dilution two-tenths cc. of the acetone dilution was added, and this mixture required 0.35 cc. of the hypochlorite. On repetition four-tenths cc. was required.

In each case ten cc. of the iodine and soda solution was used and all other conditions were kept fairly uniform.

In the case wherein the hypochlorite was added to alcohol alone no precipitate nor cloudiness was visible, although 0.1 to 0.125 cc. was required to obtain the starch reaction. When acetone had been added to the alcohol one-half this quantity of the hypochlorite was sufficient to give decided cloudiness.

These results appear to confirm the conclusions of Robineau and Rollin to the effect that the presence of ethyl alcohol has no effect upon the titration of acetone by this method, although ethyl alcohol is an iodoform-yielding substance. The small quantity of hypochlorite required to obtain the starch reaction when alcohol alone was titrated was probably in consequence of traces of impurity in the alcohol.

THE DETERMINATION OF SULPHUR IN CAST IRON.

By Francis C. Phillips.

Received November 10, 1896.

IN a paper read before the American Chemical Society in August, 1895,[1] I have detailed some experiments made in the determination of sulphur in white cast iron by the evolution method, and have attempted to show that the loss of sulphur in its

[1] This Journal, 17, 891.

determination in such iron may be due to the formation of organic sulphur compounds not oxidizable to sulphuric acid by the usual means.

By passing the gases evolved during the solution of the iron in hydrochloric acid through a heated porcelain tube it was found that the volatile organic sulphur compounds may be decomposed and nearly all the sulphur recovered by conversion into hydrogen sulphide, oxidation and precipitation as barium sulphate.

In judging of the correctness of an analytical method it has been necessary in the case of the majority of the constituents of iron to depend upon a single criterion; that method is regarded as most accurate which, being correct in its details, yields the highest percentage of the constituent sought to be determined. For it is hardly possible to add to pure iron a known percentage of sulphur, phosphorus or carbon, and test the method by a determination of the added constituent. For the determination of sulphur in iron it has been common to regard the method of oxidation and solution of the iron by nitric acid, followed by precipitation of the sulphur in form of barium sulphate as the most accurate, inasmuch that it yields results somewhat higher than those obtained by other modes of procedure.

It does not seem probable that an appreciable error could occur in the use of this method unless, in the simultaneous oxidation of the carbon and sulphur of the iron, an organic sulphur compound should be formed.

It has seemed to be of interest, however, to apply a method for the determination of sulphur by which all the constituents of the metal could be completely oxidized in a dry state and at a high temperature, in order to avoid as effectually as possible the chances of loss due to the conversion of sulphur into a volatile compound not oxidizable by ordinary means to sulphuric acid.

In searching for a method which should answer these requirements, it seemed possible that by heating the iron in the form of fine powder in presence of a mixture of alkaline carbonate and nitrate the sulphur might be oxidized directly and completely to the condition of a sulphate without affording an opportunity

for the escape of a trace of sulphur in some intermediate volatile or soluble compound. Accordingly an experiment was tried in the following way:

An iron containing its carbon in the combined form was melted in a crucible and poured while fused into water. The granulated metal was crushed in a steel mortar to an extremely fine powder. The powder so obtained was sifted through bolting sheeting.

Two and one-half grams of the sifted iron were mixed with ten grams of a mixture of equal parts of sodium nitrate and carbonate in a platinum crucible. The crucible was covered and heated over a Bunsen burner. At a red heat a sudden and rather violent reaction occurred, and having been begun, was easily maintained with very little aid from the burner flame. The reaction appeared to be complete in a few minutes. After heating for a half hour the crucible was cooled and its contents softened in water. A residue of a reddish brown powder, consisting of ferric oxide with a little ferrous oxide, was obtained. This residue was found to contain no sulphuric acid, and on digesting with hydrochloric acid dissolved without effervescence, showing that none of the particles of the original iron had remained unoxidized. From the results of this experiment and others which need not be detailed here, it seemed to be possible to oxidize finely divided iron so completely by heating with sodium carbonate and nitrate, that its sulphur might be converted quantitatively into sulphuric acid.

The mixture of sodium carbonate and nitrate although tending to oxidize finely divided iron, seems to exert a less powerful action upon the carbon contained in the iron, and this carbon may appear as a black residue after the fused mass has been softened and extracted by water and the ferric oxide dissolved in hydrochloric acid.

It seems to be important for the success of the method that in the oxidation of the iron the carbon should also be nearly or completely oxidized, for if the carbon remained unburned a portion of the sulphur might escape oxidation. In general it may be said that the order of oxidation of these three elements by the method used is as follows: 1, iron; 2, carbon; 3, sulphur; the

iron being the most easily oxidized, and the sulphur the most difficult to oxidize. This order is not exactly what we should anticipate, but it is to be remembered that unless the iron grains are fine enough to be penetrated by oxygen, and changed completely into a soft powder of ferric oxide, the sulphur and carbon have no opportunity to oxidize at all. If the iron could be used as an impalpable powder the order of oxidation would probably be different. The marked resistance of the carbon to oxidation has been frequently observed, even when using more sodium nitrate in the fusion than is theoretically enough to completely oxidize both iron and carbon, supposing that the sodium nitrate is reduced only to nitrite in the process.

Experiments of a similar kind were tried with ferromanganese. A metal containing about eighty per cent. of manganese was used. By crushing in a steel mortar this iron was very easily reduced to a powder fine enough to pass through bolting sheeting. On heating the powder with the mixture of sodium nitrate and carbonate a most violent reaction occurred, the metal burning with a long flame, extending several inches above the crucible. In order to control the reaction it was found necessary to melt one-half of the fusion mixture to be used in the crucible and then add slowly the other half, previously mixed with the powdered metal, while stirring constantly. In this way the reaction could be easily controlled. On softening the fused mass in water it was found that the iron had been peroxidized and the manganese changed to binoxide. No trace of sodium manganate was ever formed, the solution in water being after filtration invariably colorless. No carbon was found in the residue. The oxidation of the carbon is much more easily effected in the case of iron containing a high percentage of manganese. In all the trials made the silicon of the iron was oxidized, but it was found that when the fused mass is softened in water very little silica enters into solution as an alkaline silicate, the greater portion remaining insoluble and in a flocculent form.

Experiments were then tried with a gray iron. This form of iron could not be crushed to a fine powder, and an experiment was made in reducing it from small drillings by means of a

chilled iron rubber and plate, such as is ordinarily used for grinding ores. Several gray irons were tried in this way. Some could not be powdered by the method just mentioned, the grains tending to flatten instead of being crushed. Others were readily reduced, but the powder was not in any case fine enough for sifting through bolting sheeting. It was found in the case of a gray iron reduced to powder by the method of grinding, that on fusion with the mixture of sodium nitrate and carbonate, used in the preceding experiments, the graphitic carbon of this iron was more readily burnt than the combined carbon of white iron.

As it had proved to be a somewhat difficult matter to oxidize completely the carbon of the iron in the various experiments made with the fusion method, notably in the case of white iron, some trials were made in the use of sodium peroxide. This proved to be a more efficient oxidizing agent for iron and its contained carbon than sodium nitrate. For these trials a mixture was used consisting of forty-five parts each of sodium peroxide and sodium nitrate, together with ten parts of sodium carbonate.

White iron was oxidized and its carbon burnt during a fusion lasting less than ten minutes.

On heating ferromanganese with this mixture the iron was found to be completely oxidized. The carbon was burnt and the manganese was oxidized and converted into sodium manganate, yielding a deep green solution when the fused mass was digested in water.

An admixture of sodium carbonate to sodium peroxide tends in all cases to diminish its action upon finely divided iron at a high temperature and renders the process more easily controlled. It seemed to be possible to base a method for the quantitative determination of sulphur in certain kinds of cast iron upon the reactions described above.

An indispensable condition of success in the use of the method is found in the extreme fineness of the iron. In the case of white irons the fineness of the powder has been secured by

crushing in a steel mortar until the powder passed through a sieve of bolting sheeting or bolting cloth.[1]

Some gray irons cannot be crushed or ground. To these the method is not applicable. For gray irons, however, the evolution method answers all requirements.

The following details are given of the method finally employed:

1. *White iron.*—About one and one-half grams of the finely powdered and sifted metal was intimately mixed with eight grams of the sodium peroxide mixture above mentioned, or with four grams each of sodium carbonate and nitrate. The somewhat violent reaction set up on the application of strong heat to the platinum crucible was completed in a few minutes. The crucible was heated for about twenty minutes in all. After cooling the contents were softened in water, the solution decanted and the residue ground, while wet, in a mortar. The solution and residue were then digested in a beaker on the water-bath for one hour after addition of two cc. of strong bromine water. The liquid was then filtered, acidulated with hydrochloric acid, evaporated to dryness to separate the small portion of silica which had entered in solution and filtered. The sulphuric acid was determined in the filtrate in the usual manner. The barium sulphate obtained was always white. If the fusion mixture contains sodium carbonate and nitrate, but no sodium peroxide, the crucible must be heated for a longer time, but a portion of the carbon of the iron may still remain unoxidized.

2. *Ferromanganese.*—In this case it is better to use a mixture of equal parts of sodium nitrate and carbonate, omitting the sodium peroxide.

Ten grams of the mixture were divided into two portions, one of which was fused in a crucible. The other portion mixed with two or two and one-half grams of the finely powdered iron

[1] Two different materials are sold which are suitable for the sifting. One is called bolting cloth, the other bolting sheeting. The bolting cloth used in these experiments contained about eighty-five meshes to the linear inch, while in the bolting sheeting about one hundred and thirty-five were counted. The material having the smaller number of meshes is made of coarser threads, however, and yields, on account of the smaller openings, a finer powder. Bolting cloth is, on this account, better suited to the preparation of a sample of white iron for a determination of sulphur by the method described.

was then slowly added. Although too violent combustion of the iron is to be avoided, it seems to be important, for the success of the method, that a reaction of decided intensity should occur during the fusion.

Sodium nitrate possesses an advantage over sodium peroxide in its greater purity, the former compound being readily obtainable with practically insignificant traces of sulphur.

Natural gas was the fuel used for the Bunsen burner in heating the charges. This gas was found by repeated experiments, not to contain a sufficient quantity of sulphur to affect the purity of the sodium carbonate when heated in a platinum crucible in the same manner as in the case of the determinations described.

The usual occurrence of sulphur compounds in coal gas would preclude its use in the application of the method.

From the experiments, the results of which are stated in the accompanying table, there seems to be some reason to suppose that not quite all the sulphur of the iron is converted into barium sulphate when the metal is oxidized and dissolved by nitric acid. That it has been completely recovered by the process of fusion cannot be positively asserted.[1]

The method I have described is not proposed as a substitute for any existing method. The purpose of the present work was merely to ascertain as far as possible whether by a process of direct oxidation of the iron in a dry state a larger proportion of the sulphur could be recovered in weighable form than by the usual method of oxidation and solution in nitric acid.

My thanks are especially due to Mr. F. B. Smith for great care and attention to detail in conducting the experiments I have detailed.

[1] The method of preparation of a sample for analysis in the case of the more brittle forms of iron, by crushing in a steel mortar and sifting, is suggested in Regnault's Elements of Chemistry, translated from the French by Betton, 1867, 2, 112.

Character of iron used.	Fusion mixture employed.	Percentage of sulphur found by fusion.	Percentage of sulphur found by the method of oxidation by nitric acid.
White iron A crushed in mortar and sifted through bolting sheeting.	Contained equal parts of sodium carbonate and nitrate.	0.112	0.101
		0.112	0.098
		0.111	0.096
		0.107	0.099
		0.114	0.100
		0.114	0.102
		0.106	0.102
		0.108	0.104
		0.107	
		0.103	
Means		0.109	0.100
White iron B crushed and sifted.	Contained 45 parts $NaNO_3$ 45 parts Na_2O_3 10 parts Na_2CO_3	0.155	0.143
		0.150	0.149
		0.130	0.143
		0.139	0.147
		0.166	
		0.156	
		0.156	
		0.161	
		0.151	
Means		0.151	0.145
Ferromanganese crushed and sifted.	Contained equal parts of sodium nitrate and carbonate.	0.022	0.012
		0.027	0.013
		0.018	0.012
		0.018	0.010
		0.018	
		0.019	
		0.016	
Means		0.020	0.012
Gray iron drillings powdered by rubber and plate. Not sifted.	Contained equal parts of sodium nitrate and carbonate.	0.034	0.027
		0.030	0.030
		0.036	0.026
		0.034	0.028
		0.033	0.028
		0.034	0.022
Means		0.033	0.027

CARBON DETERMINATIONS IN PIG IRON.

By Bertrand S. Summers.

Received October 3, 1896.

THOSE chemists who have had occasion to do many carbon determinations in pig iron, to which was allotted but little time, have probably felt the need of improvements in some of our standard methods.

The old oxygen combustion method, although accurate, requires more time than can usually be spared if use is made of a porcelain or glass tube. However, it has the greatest of all advantages, that of accuracy. The writer has used for some time a regular Bunsen furnace with a glass tube, and while the results were all that could be desired, the time required for a refractory residue was almost three hours.

A series of experiments was conducted with the ordinary chromic acid process, but the results were quite unsatisfactory. Every precaution was taken to insure accuracy, but with high carbon residue low results were obtained in nearly every case when checked by the oxygen combustion method. This was particularly noticeable when a considerable content of graphite was present. The results checked quite well with each other and gave satisfactory results when working on steel.

As this state of affairs greatly embarrassed matters in the laboratory, an effort was made to devise some means by which the carbon could be determined with reasonable speed and accuracy.

Recognizing the advantages of the combustion method, it was decided to make use of a platinum tube. To avoid delay and expense the tube was manufactured in the factory. It was made of 0.200 stock twelve inches long and eleven-sixteenths inch in diameter. A perfectly tight tube was constructed by using ordinary gold solder, which may be obtained from any jeweler. Around each end of the tube copper coolers were brazed, in order to cool the tube in the neighborhood of the rubber stoppers. The inlet of the coolers served the double purpose of supports and water supplies. In spite of this precaution it was found that the air circulating through the heated portion of the tube was hot enough, on reaching the stoppers, to seriously affect them. In order to prevent this, the scheme shown in Fig. 3

was devised. The funnel shape protuberance here seen was filled with ignited asbestos, and the whole was removed with the stoppers. This appliance proved an effectual preventive for further heating of the stoppers, as a red heat could be maintained two inches from them and they remain perfectly cool.

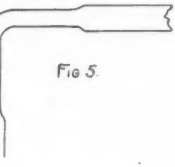


Fig. 5.

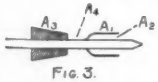


Fig. 3.

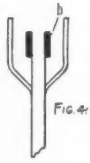


Fig. 4.

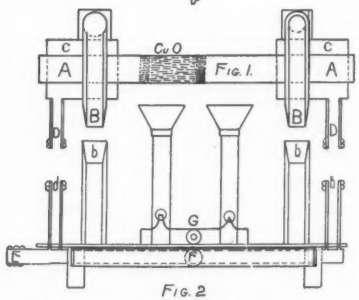
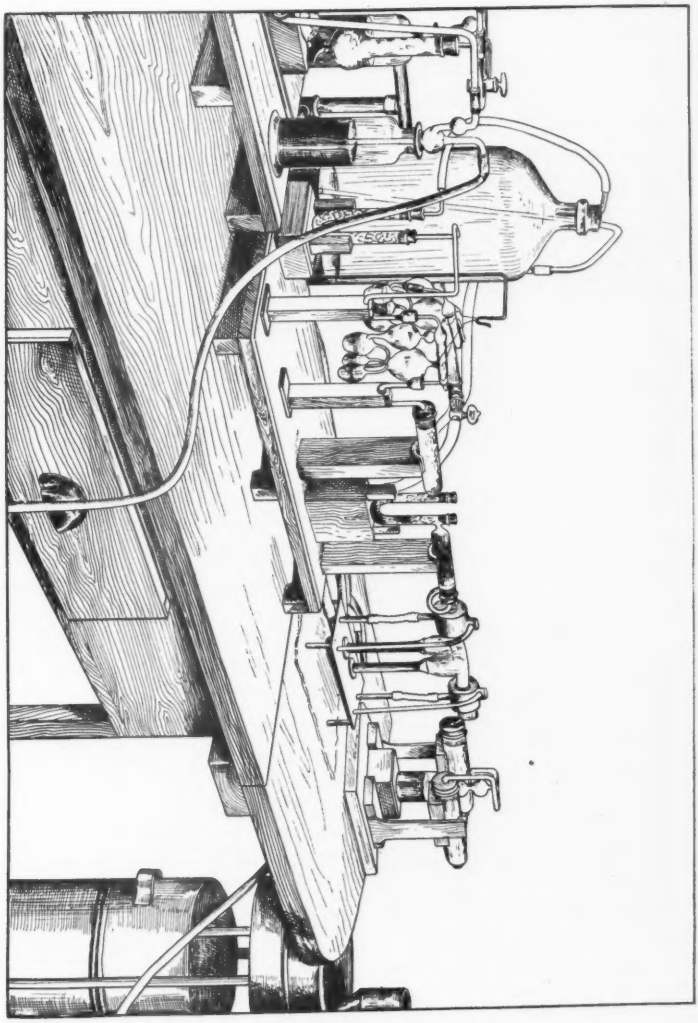


Fig. 2

Fig. 1—AA, Platinum tube; BB, Support and water outlet; CC, Coolers; DD, Water supplies.
Fig. 2—bb, Sockets for BB; dd, Connection for water supply, DD; E, Main water supply; F, waste pipe; G, Gas connection.
Fig. 3—A_3, Stoppers; A_1, Glass cup for asbestos, A_2; A_4, Outlet.
Figs. 4 and 5—Showing connections for mercury joint.

With this arrangement it was found that a high carbon residue could be burned completely in twenty minutes. It became evident from this that if the aspirating space were decreased, good results could be obtained in a comparatively short time. With this idea in view, the train depicted in the accompanying picture was designed and made by our own glass-blowers. The train has the further advantage that rubber connections are avoided, the only rubber tubing in use being at the ends of the combustion tube.

The purifying train consists of a large U-tube of one and one-half inch stock and twelve inches long. The first limb is filled with broken caustic potash, and the second with fused calcium chloride. The first limb connects with a Drechsel bottle partially filled with strong sulphuric acid, and the second with the combustion tube.



The purifying train on the absorption end is made in one piece. It consists of a five inch U-tube of thick walled glass five-eighths inch in diameter, into the sides of the limbs of which are fused arms. These arms are made of one-inch stock and about seven inches long. The first arm is filled with anhydrous cuprous chloride and anhydrous cupric sulphate. The U-tube serves as the receptacle for the sulphuric acid, and the second arm is filled with calcium chloride previously treated with an excess of carbon dioxide.

The connection with the Geissler bulbs is established by means of mercury joints. These serve to facilitate removal of the bulbs and make a joint which is perfectly secure. The joint can readily be made by any glass blower, an illustration of which is seen in Fig. 4. The end of the Geissler bulb is so reamed as to fit loosely over the tube inside the cup (Fig. 5). A small piece of rubber tubing (*b*, Fig. 4) is slipped over the tube and makes a moderately tight joint with the end of the Geissler bulb. When the cup is filled with mercury a perfect connection is obtained. The method of connecting the Geissler bulb with rubber tubing was both awkward and liable to leakage. These junctions have been in use for some time in our laboratory and have given thorough satisfaction.

With this apparatus as described the most refractory residues are burned in an hour and a half. With residues of less refractory nature and lower carbon content an estimation may be completed in less time. The blank on the apparatus never exceeds three-tenths of a milligram, and is usually one-tenth or nil.

Some results are here appended, thinking they may be of interest. Those obtained by the chromic acid process were quite scattering unless great care was exercised and sufficient time was allowed. The results from this method, given below, are those where much time was given and great pains taken to insure complete oxidation. Values from the Bunsen furnace are given to serve for comparison.

Chromic Acid Method. Total Carbon.	Bunsen Furnace. Total Carbon.
3.23	3.31
3.27	3.33
3.23	...
3.28	...

Results from the above described process, when compared with the Bunsen furnace, were very good.

Platinum Furnace. Total Carbon.	Bunsen Furnace. Total Carbon.
3.03	3.03
3.03	3.05
3.05	...

The convenience of this apparatus in expediting work in the laboratory has led me to write this description, in the hope that it might be of service to other chemists.

CHEMICAL LABORATORY, WESTERN ELECTRIC COMPANY,
CHICAGO.

NOTE ON THE SOLUBILITY OF BISMUTH SULPHIDE IN ALKALINE SULPHIDES.

BY GEORGE C. STONE.

Received November 9, 1896.

IN the August number of this Journal there is a note by Prof. Stillman on this subject; he shows that if a solution containing bismuth is made alkaline by sodium hydroxide and then heated with an excess of an alkaline sulphide a considerable amount of bismuth is held in solution. On repeating his experiments qualitatively I obtained the same result, but when the bismuth was first precipitated as sulphide from an acid solution and then treated with an alkaline sulphide but little if any was dissolved.

To test the solubility quantitatively I made a solution of about one and two-tenths grams of bismuth hydroxide in 500 cc. of very dilute hydrochloric acid; in two portions, of fifty cc. each. I determined the bismuth by precipitation by ammonium carbonate, finding 0.0966 and 0.0965 gram.

I next precipitated the bismuth in two more lots of the same solution by hydrogen sulphide, filtered and heated the precipitate for half an hour with a large excess of potassium sulphide, filtered, dissolved and reprecipitated by ammonium carbonate, the bismuth weighed 0.0981 and 0.0970 gram.

Two more lots treated in the same manner, except that they were heated with ammonium sulphide, gave 0.0970 and 0.0976 gram of bismuth.

From the above it seems fair to conclude that bismuth sulphide precipitated from an acid solution is not dissolved by subsequent treatment with an alkaline sulphide.

[Contribution from the Laboratory of Agricultural Chemistry of the Ohio State University.]

ON THE BEHAVIOR OF COAL-TAR COLORS TOWARD THE PROCESS OF DIGESTION.

By H. A. Weber.

Received October 10, 1896.

IT is very well known that the coal-tar colors have come into general use for coloring confectionery and other articles of food and drink. In fact they have almost completely superseded the vegetable colors, which have been used from time immemorial for a similar purpose. The indiscriminate use of these colors, some of which are derived from bodies of a decidedly poisonous nature, has often been regarded with suspicion by persons who are interested in public health. On account of the uncertainty existing in regard to these colors from a sanitary point of view, Austria has prohibited their use *in toto* in all articles of food and drink. Other countries prohibit certain of the colors, which have been shown to be injurious, and allow all others to be used.

The experiments made upon the lower animals have, in the main, revealed negative results. Thus the writer about eight years ago fed some of the colors most commonly employed by confectioners to rabbits in order to test this question. One-half gram of the colors, among which magenta and corallin were included, was fed to as many rabbits per day for ten days in succession without any apparent ill effects. The exhaustive treatise of Dr. Weil, translated by Leffmann, ascribes toxic effects to only a small number of the many colors employed by him in his experiments upon domestic animals.

The effect which these colors might exert upon digestive ferments, however, was a subject which had as yet received no attention, and the following experiments were undertaken in order to throw some light upon this question. The ferments employed were Armour's pepsin and pancreatine, liberal samples of which were kindly furnished by Armour & Co., of Chicago.

For the purpose of showing the digestive action, blood fibrin preserved in alcohol was employed. The fibrin was soaked and

thoroughly washed with water to remove the alcohol, then pressed between filter paper, and the amount required for each experiment weighed off.

In each set of experiments a control experiment was carried on without the addition of color. The mixture was made as follows:

Hydrochloric acid solution (two-tenths per cent.)	100 cc.
Pepsin	20 milligrams.
Fibrin	1 gram.

This mixture placed in a large test-tube was digested in a water-bath at a temperature of 38° to 40° C. until the fibrin was dissolved.

At the same time similar mixtures as above containing in addition 1, 0.5, 0.250, 0.125, and 0.062 gram of the color to be tested respectively, were digested in the same water-bath for the time required to dissolve the fibrin in the control experiment. Any fibrin remaining undissolved in the latter tests, was removed, thoroughly washed, pressed between filter paper as before and weighed.

1. PEPSIN AND OROLINE YELLOW.

This color was one of a series employed in the coloring of confectionery, and was found to be what is known in the trade as *Acid Yellow* or *Fast Yellow*, and is a mixture of sodium amidoazobenzenedisulphonate with sodium amidoazobenzenemonosulphonate.

	Amount of color. Gram	Amount of fibrin. Gram.	Amount of pepsin. Gram.	Duration of experiment. Hours.	Amount of fibrin dissolved. Gram.
1	0.0	1	0.020	3	1.0
2	1.0	1	0.020	3	0.1
3	0.5	1	0.020	3	0.12
4	0.25	1	0.020	3	0.22
5	0.125	1	0.020	3	0.35
6	0.062	1	0.020	3	0.73

From this it will be seen that even in test No. 6, where the color employed amounted to only one part in 1600 parts of the solution, the presence of the color had still a depressing effect. For fear that, owing to the nature of this color, the hydrochloric acid might have been neutralized in part, the experiment was

repeated with a six-tenths per cent. solution of hydrochloric acid with similar results.

Of course the determination of the fibrin dissolved is only approximate, as can readily be inferred from the way it was done.

In tests Nos. 2, 3 and 4 no change in the amount of fibrin was apparent to the eye. That a small part of the fibrin had gone into solution was confirmed by the fact that a slight precipitate of albuminoids was obtained on the addition of a solution of tannin. On the whole it must be conceded that this color has a marked and injurious effect upon peptic digestion.

2. PEPSIN AND SAFFOLINE.

This is also a candy color and was found to be *acridine red*.

Amount of color. Gram.	Amount of fibrin. Gram.	Amount of pepsin. Gram.	Duration of experiment. Hours.	Amount of fibrin dissolved. Gram.
0.0	1	0.020	$3\frac{1}{2}$	1
1.0	1	0.020	5	1
0.5	1	0.020	5	1
0.25	1	0.020	5	1
0.125	1	0.020	$3\frac{1}{2}$	1
0.062	1	0.020	$3\frac{1}{2}$	1

As will be seen from the table above, this color only slightly retards the digestion of the fibrin in the three stronger solutions, while in the last two tests there was no interference with the process. On the whole it may be said that the effect of this color on peptic digestion is practically nil.

3. PEPSIN AND MAGENTA.

It is needless to tabulate the results of this experiment. Suffice it to say that the solution of the fibrin in the five tests containing the same proportions of the color as employed above kept pace throughout the whole duration of the experiment with the control test, the fibrin in all cases dissolving at the expiration of three and one-half hours.

This color, therefore, seems not to interfere with peptic digestion.

These four colors were also employed with pancreatin, and the method was as follows:

For the control experiment the following mixture was made :

Water	100 cc.
Sodium bicarbonate	1.5 grams.
Pancreatin	0.3 gram.
Fibrin	1.0 gram.

This mixture contained in a large test-tube, was digested in a water-bath until the fibrin was peptonized. To test the effect of the colors, there was added to similar mixtures as above 1, 0.5, 0.25, 0.125 and 0.062 gram of each color respectively.

5. PANCREATIN AND ÓROLINE YELLOW.

To the great surprise of the writer, this color, which had proved so effective in stopping and retarding peptic digestion, was found to exert no action whatever on the pancreatic ferment ; the fibrin in all five of the tests with this color, dissolved as freely as that of the control test. The solution of the fibrin in all cases was completed at the expiration of six hours.

PANCREATIN AND SAFFOLINE.

The action of this color was quite different from that of oroline yellow, as the subjoined table will show :

	Amount of color. Gram.	Amount of fibrin. Gram.	Amount of Pancreatin. Gram.	Duration of experiment. Hours.	Amount of fibrin dissolved. Gram.
1	0.0	1	0.3	6½	1.0
2	1.0	1	0.3	6½	0.0
3	0.5	1	0.3	6½	0.0
4	0.25	1	0.3	6½	0.55
5	0.125	1	0.3	6½	0.65
6	0.062	1	0.3	6½	0.75

These results show, that in the two stronger solutions the action of the pancreatic ferment was entirely stopped, and that even in test No. 6, which contained only one part of color to 1600 of the solution the action of the ferment was retarded to a marked extent.

Tannin precipitates the coloring matter.

7. PANCREATINE AND MAGENTA.

This color was as marked in retarding and stopping the action of pancreatine as saffoline. The results are given in the table below :

	Amount of color. Gram.	Amount of fiber. Gram.	Amount of pancreatine. Gram.	Duration of experiment. Hour.	Amount of fiber dissolved. Gram.
1	0.0	1	0.3	6½	1.0
2	1.0	1	0.3	6½	0.0
3	0.5	1	0.3	6½	0.0
4	0.25	1	0.3	6½	0.40
5	0.125	1	0.3	6½	0.60
6	0.062	1	0.3	6½	0.73

The solutions of tests 2 and 3 gave no precipitate with tannin. In all other tests the precipitate was either marked or heavy.

8. PANCREATINE AND METHYL ORANGE.

This color in all of the tests behaved like the last three colors described, completely stopping the action of the pancreatine in the two strongest solutions and retarding it to a marked extent in the weakest. The tabular statement would be similar to the last.

It seems then, so far as these four colors are concerned, that none interfere with both peptic and pancreatic digestion, but that each color interferes seriously with either the one or the other. What the action of other coal tar colors may be, can, of course, not be inferred from this limited number of experiments, but it may safely be said that bodies which have such a decided action in retarding the most important functions of the animal economy, cannot properly have a place in our daily food and drink.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, NO. 19.]

THE ACTION OF ACID VAPORS ON METALLIC SULPHIDES.

BY JEROME KELLEY, JR. AND EDGAR F. SMITH.

Received October 2, 1896.

EXPERIMENTS made in this laboratory on the action of the vapors of hydrochloric acid upon the sulphide of arsenic proved that the latter is wholly volatilized. The purpose of the present communication is to record further observations along analogous lines. Thus, when washed and dried arsenic trisulphide is exposed to the action of hydrobromic acid gas, it volatilizes completely. Indeed the action commences in the cold with the formation of a liquid that passes out of the containing

vessel upon the application of a very gentle heat. In evidence of this, two quantitative experiments may be given :

Arsenic sulphide taken. Gram.	Arsenic sulphide expelled. Gram.
0.2945	0.2941
0.4632	0.4628

Antimony trisulphide, like that of arsenic, is volatilized by hydrochloric acid gas. It was quite probable that a like deportment would be observed if hydrobromic acid gas should be substituted. This was found to be the case. When the gas came in contact with the sulphide it became liquid and volatilized as soon as a gentle heat was played upon the boat in which the sulphide was contained.

Antimony sulphide taken. Gram.	Antimony sulphide expelled. Gram.
0.1473	0.1469
0.0938	0.0935

Upon substituting stannic sulphide for antimony sulphide, an experience similar to that observed with antimony and arsenic sulphides followed. There was a complete volatilization with but a trifling residue, which proved to be carbon from filter paper that had adhered to the metallic sulphide.

Stannic sulphide taken. Gram.	Stannic sulphide expelled. Gram.
0.1880	0.1880
0.5527	0.5521.
0.4174	0.4169

The oxides of arsenic, antimony and tin (at least in the stannic form) can be volatilized in a current of hydrochloric acid gas. This is also true of the sulphides of arsenic and antimony, but how the two sulphides of tin would act under like conditions was not known.

Experiments recently made demonstrate the perfect volatility of stannic sulphide in this way. With stannous sulphide it was found that by the continued action of the gas in the cold there followed a complete conversion into chloride without any volatilization. That the residue was the chloride was evident from its action upon a mercuric salt solution. The figures obtained in the several trials were :

Stannous chloride found. Gram.	Stannous chloride theory. Gram.
0.3544	0.3523
0.4893	0.4903

Several attempts were made to separate stannous and stannic sulphides by this procedure. The results were unsatisfactory. In order to drive out the stannic salt completely it is necessary to heat the mixture, and this caused a partial volatilization of the stannous chloride, so that quantitative results could not be obtained.

Comparatively few metallic sulphides have been studied in the direction indicated in the preceding lines, so that it is probable a wider application of the method will disclose interesting behaviors, and that probably new separations can be brought about in this way. The action of the vapors of haloid acids has also been tried on natural sulphides with a fair degree of success.

[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, NO. 20.]

TUNGSTEN HEXABROMIDE.

BY HERBERT A. SCHAFFER AND EDGAR F. SMITH.

Received October 10, 1896.

THE most recent work upon tungsten bromides is that of Roscoe,[1] who endeavored to prepare a hexabromide, but obtained instead a penta derivative from which the dibromide was subsequently made. By reference to the literature bearing upon this subject it will be noticed that bromine, diluted with carbon dioxide, was made to act upon tungsten metal exposed to a red heat. Experimental evidence is at hand that tungsten at high temperatures deoxidizes carbon dioxide, thus allowing ample opportunity for the production of oxybromides, which, in spite of the greatest care, are sure to appear in larger or smaller amount. The thought also suggested itself that possibly the "red heat" at which the action was allowed to occur might have been detrimental and have indeed prevented the formation of the hexabromide.

Hence, we determined to operate in an atmosphere of nitro-

[1] *Ann. Chem.* (Liebig), 162, 362.

gen and to apply a very gentle heat to the vessel containing the tungsten. In this connection it may be mentioned that the nitrogen was conducted through a series of vessels charged with chromous acetate, sulphuric acid, caustic potash, and phosphorus pentoxide, respectively. It then entered an empty vessel into which dry bromine was dropped from a tap-funnel, and after passing through a tall tower, filled with calcium chloride, entered a combustion tube resting in a Bunsen furnace. The anterior portion of the combustion tube was contracted at intervals, forming a series of bulbs, and at its extremity was connected with an empty Woulff bottle, followed by a calcium chloride tower, and finally a receiver filled with soda lime and broken glass. A steady current of nitrogen was conducted through this system for a period of three days. On the fourth day bromine was introduced. The tungsten contained in the combustion tube was heated very gently. In a short time brown vapors appeared. These condensed to a liquid beyond the boat and eventually passed into blue-black crystalline masses that separated from the walls of the tube, when perfectly cold, with a crackling sound. Very little heat was required to melt them and they could with care be resublimed in distinct, blue-black needles. The latter was collected in one of the bulbs (No. 2) previously mentioned. Other products were observed and isolated. All were analyzed. Bulb No. 1—that nearest the tungsten metal—contained a black, velvety compound, which upon analysis showed the presence of tungsten dibromide. Bulb No. 2 contained 0.2103 gram of the blue-black crystals, which yielded 0.0577 gram of tungsten, or 27.43 per cent., and 0.1543 gram of bromine, or 73.53 per cent. The theoretical requirements of tungsten hexabromide are 27.72 per cent. tungsten and 72.28 per cent. bromine. The bromine percentage found is high. This may be due to traces of bromine that had not been driven out from the crystalline deposit, or to adherent silver tungstate, as some tungstic acid remained in the solution from which the silver bromide was precipitated.

A fresh portion of the blue-black crystals was prepared as before and analyzed. The bromine determination was unfortunately lost. The determination of the tungsten resulted as fol-

lows : 0.4351 gram of material gave 0.1222 gram of tungsten or 28.08 per cent.

A third preparation was made. On subjecting 0.1775 gram of it to analysis these results were obtained :

0.0496 gram tungsten or 27.94 per cent.

0.1266 gram bromine or 71.32 per cent.

Tabulating the series, we have :

		Found.		Mean	Required for hexabromide.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Tungsten.....	27.43	28.08	27.94	27.81	27.72
Bromine.....	73.53	71.32	....	72.33	72.28

These figures give evidence that the body analyzed is tungsten hexabromide.

In analyzing the third portion of the blue-black needles the bromine was determined by placing the material in a small Erlenmeyer bulb, covering it with nitric acid and then distilling. The liberated bromine was passed into a silver nitrate solution.

The tungsten hexabromide prepared by us consists, as already observed, of blue-black needles. Moderately elevated temperatures decompose the compound. It gives off fumes when brought in contact with the air. Water decomposes it with the formation of a royal-blue colored oxide. Ammonia water dissolves it, the solution remaining colorless. A vapor density determination resulted negatively, as decomposition was apparent early in the experiment.

NOTES ON THE FERROCYANIDES OF ZINC AND MANGANESE.

By Edmund H. Miller.

Received October 10, 1896.

THE composition of the ferrocyanides of zinc and manganese, formed when salts of these metals are precipitated by potassium ferrocyanide, is given by Prescott and Johnson[1] as $Zn_2Fe(CN)_6$ and $Mn_2Fe(CN)_6$, while the books on volumetric analysis, such as Sutton's and Beringer's, ignore the composition of this precipitate.

[1] Qualitative Analysis, pages 67 and 57.

The prevailing idea is that in the titration of zinc by potassium ferrocyanide, a normal zinc ferrocyanide is formed. This I believe to be incorrect, for if the reaction is

$$K_4Fe(CN)_6 + 2ZnCl_2 = Zn_2Fe(CN)_6 + 4KCl,$$

a solution of potassium ferrocyanide, one cc. of which is equivalent to ten milligrams of zinc, would contain 32.32 grams of $K_4Fe(CN)_6.3H_2O$ to the liter, not 43.2[1] to 45[2] grams, as has been found by experiment. Using forty-four grams per liter as a basis for calculation, the reaction becomes

$$2K_4Fe(CN)_6 + 3ZnCl_2 = Zn_3K_2(Fe(CN)_6)_2 + 6KCl.$$

This reaction is not merely one that may possibly be true, but according to Wyrouboff,[3] the precipitate formed by the action of potassium ferrocyanide on a zinc salt, whichever is in excess, is $3Zn_2Fe(CN)_6.K_4F_2(CN)_6.12H_2O$, white, while the normal salt, $Zn_2Fe(CN)_6.4H_2O$, is formed only by the action of hydroferrocyanic acid on a zinc salt.

This statement agrees both with the preceding reaction and with the results obtained in standardizing potassium ferrocyanide solution.

The manganese precipitate with potassium ferrocyanide, as obtained in titration, is given by Stone[4] as $Mn_3Fe_2(CN)_{12}$. This is a ferri-, not a ferrocyanide, thus making necessary a change of quantivalence. Mr. Stone also states that an amount of potassium ferrocyanide which will precipitate four atoms of zinc will only precipitate three of manganese, thus basing his calculation on the formation of a normal zinc ferrocyanide.

Wyrouboff[1] gives the precipitate obtained from potassium ferrocyanide and a manganese salt, whichever is in excess, as

$$5Mn_2Fe(CN)_6.4K_4Fe(CN)_6.4H_2O, \text{ rose white;}$$

while the normal salt $Mn_2Fe(CN)_6.7H_2O$, cream, is formed as in the case of zinc by hydroferrocyanic acid.

The solution used by Mr. Stone had the following strength:

1 Sutton: Volumetric Analysis, p. 329; Beringer: Assaying, p. 219.
2 Furman: Assaying, p. 205.
3 *Ann. chim. phys.*, [5], 8. 485.
4 *J. Am. Chem. Soc.*, 17. 473.

One cc. = 0.00606 gram zinc.
One cc. = 0.00384 gram manganese.

If the ratio were exactly four zinc to three manganese, using the most recent atomic weights, the strength of this solution against manganese would be one cc. = 0.00382 gram, while, according to Wyrouboff, $10Mn = 9K_4Fe(CN)_6$ and $6Zn = 4K_4Fe(CN)_6$, or $10Mn = 13.5$ Zn, or $1Mn = 1.35$ Zn, and the strength against manganese would be 1 cc. = 0.003774 gram.

These figures show but little difference between the two ratios and while Mr. Stone's experimental results are undoubtedly accurate, this theory based on the formation of $Zn_2Fe(CN)_6$ and $Mn_3Fe_2(CN)_{12}$ is not satisfactorily proved.

This article is only a preliminary note regarding the composition of the ferrocyanides as they are being investigated in this laboratory.

In connection with the ferrocyanide of zinc I have found a very strong solution of hydrochloroplatinic acid, H_2PtCl_6, acidified with hydrochloric acid, a most satisfactory indicator for the titration of zinc by potassium ferrocyanide, when performed in a hot solution. This indicator is used in the same way as uranium acetate and is less affected by a varying amount of hydrochloric acid. The end reaction is a bright emerald green, which takes a few seconds to develop. It will not work with a cold solution.

ASSAY LABORATORY, COLUMBIA UNIVERSITY.

A MODIFICATION OF THE GUNNING METHOD FOR NITRATES.

BY JOHN FIELDS.

Received October 20, 1896.

THE full text of the official Gunning[2] method is as follows: "In a digestion flask, holding from 250 to 500 cc., place from seven-tenths to three and five-tenths grams of the substance to be analyzed, according to the amount of nitrogen present. Add thirty to thirty-five cc. of salicylic acid mixture; namely, thirty cc. sulphuric acid to one gram of salicylic acid, shake until thoroughly mixed and allow to stand five to ten

[1] *Ann. chim. phys.*, [5], 8, 474.
[2] Bulletin 46, U. S. Dept. of Agr., p. 18.

minutes, with frequent shaking; then add five grams sodium thiosulphate and ten grams of potassium sulphate. Heat very gently until frothing ceases, then heat strongly until nearly colorless. Dilute, neutralize, and distil as in the Gunning method."

This method has its advantages in that no heavy metals are added, such as zinc and mercury, which sometimes interfere with the distillation. It has, however, a few disadvantages which the following modification partially overcomes. When working with some materials, there is considerable trouble due to persistent frothing, and in some cases, it has taken six hours constant attention to get the digestion safely over this point. Moreover, unless the contents of the flask are diluted while still warm, there is a tendency for the sulphates to become hard and difficult of solution.

In the modification proposed, the following reagents are necessary:

1. Chemically pure sulphuric acid.
2. Salicylic acid.
3. Potassium sulphide.

The material containing the nitrates is weighed out into a digestion flask and thirty cc. sulphuric acid containing one gram salicylic acid are added, and gently heated to facilitate the solution of nitrates and prevent frothing later. While warm, six to seven grams of potassium sulphide are added in small portions, the flask being thoroughly shaken after each addition. It is then placed over a low flame and the heat rapidly increased until the acid mixture boils. No further attention is required and the digestion is usually complete at the end of an hour. When cold, the liquid is diluted and distilled in the usual manner.

The average difference between the results on sixty samples of fertilizers containing nitrates by the official method and the proposed modification was 0.02 per cent., those by the latter being higher.

The points of difference between the modification and the official modified Gunning are the following:

1. The number of reagents used in the digestion is reduced from four to three.

2. Frothing is obviated and the operation requires no attention except turning up the lamps until full heat is secured.

3. The time of digestion is shortened.

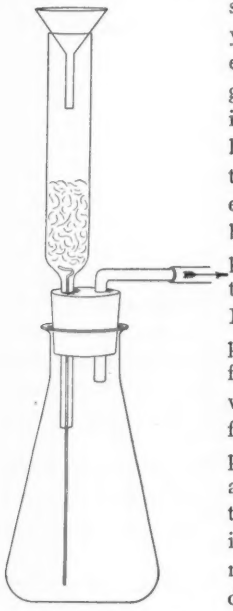
4. Potassium sulphide is made to do double work by acting as a reducing agent instead of sodium thiosulphate and then being converted into potassium hydrogen sulphate serving the end secured by adding potassium sulphate in the original method.

THE SEPARATION OF ALKALOIDAL EXTRACTS.

By Charles Platt.

Received October 20, 1896.

THE writer has found the accompanying simple device of great value in the separation of the annoying emulsions so often met with in alkaloidal analysis, as, for instance, in the petroleum ether and benzene extractions of Dragendorff's method. The filtering tube is nineteen cm. long, the upper 12.5 cm. having an inside diameter of fourteen mm., the lower contracted portion, an inside diameter of three mm. A stout platinum wire bent at the upper end is so placed as to pass through the constricted portion of the tube to the bottom of the eight-ounce Erlenmeyer flask. Washed cotton is firmly packed in the tube to a depth of about four cm. and the apparatus, connected with a filter pump, is ready for use. The filtered liquids may finally be carefully poured into an ordinary separating funnel and manipulated as usual. By this method the most persistent emulsions are separated into their constituent liquids in as many minutes as ordinarily are required hours or days.



Chemical Laboratory, Hahnemann Medical College,
Philadelphia, Pa.

THE PREPARATION OF DIETHYL MALONIC ESTER.

By W. A. Noyes.

Received October 29, 1896.

HAVING occasion recently to prepare considerable quantities of malonic ester, it has been found that the process can be very much shortened by the use of sulphuric in place of hydrochloric acid and of acid sodium carbonate in place of potassium carbonate. As the body is the starting point for a great variety of syntheses the method used may be of interest to others.

One hundred grams of chloracetic acid are placed in a porcelain dish, 21 cm. in diameter, and 200 cc. of water added. The solution is warmed and ninety grams of acid sodium carbonate added in small portions, and the warming continued until a temperature of 55°–60° is reached and effervescence nearly ceases. Eighty grams of coarsely powdered potassium cyanide is then added, and the whole stirred without further warming, till the somewhat vigorous reaction is complete. The solution is then evaporated rapidly on a thin sheet of asbestos paper till the thermometer with which it is vigorously and constantly stirred shows a temperature of 130°–135°. The hand should be protected by a glove or otherwise, and the glass of the hood in which the evaporation is conducted should be between the dish and the face during this part of the process. The mass should be stirred occasionally while cooling, and as soon as it solidifies it should be broken up coarsely and transferred to a liter flask. Add forty cc. of alcohol and connect with an upright condenser. Through the latter add, in small portions and with frequent shaking, a cooled mixture of 160 cc. of alcohol with 160 cc. of concentrated sulphuric acid. The whole may be added within five or ten minutes (instead of the day and a half required to saturate with hydrochloric acid by the old method). Toward the close there is a considerable evolution of hydrochloric acid. Heat on a water-bath for an hour. Cool quickly under the tap, with shaking to prevent the formation of a solid mass of crystals. Add 200 cc. of water, filter, wash the undissolved salts with about fifty cc. of ether, shake up with the filtrate and separate. Add a solution of sodium carbonate and shake carefully

with the ethereal solution till alkaline. Separate again, distil off the ether and dry by heating for fifteen minutes on a water-bath under diminished pressure, using a capillary tube as for vacuum distillations. The residue gives, after one distillation, an almost pure malonic ester.

The sodium carbonate solution appears to contain some of the acid ester. If this solution is added to the first acid solution, the ester separates with some ether. The ethereal solution may be separated, the ether evaporated at a gentle heat, and the residue added to the contents of the flask in which a second saponification of the cyanacetate is to be effected. If this is done, a yield of malonic ester equal to the weight of chloracetic acid used can be obtained. This is ten to fifteen per cent. better than by the old method.

ROSE POLYTECHNIC INSTITUTE,
TERRE HAUTE, IND., Oct. 27, 1896.

NOTE.

Untaxed Alcohol for Use in Manufacturing and in the Arts.—The Joint Select Committee, created at the last session of Congress, to investigate and report upon the question of the use of alcohol free of tax in the manufactures and arts, have prepared a series of interrogatories, which will be distributed throughout the country to such parties as are thought to be interested in the question.

The report of Mr. Henry Dalley, Jr., who was commissioned to investigate the workings of foreign laws governing the use of untaxed alcohol in the manufactures and arts has been submitted, and contains very full and extremely valuable data covering Great Britain, Germany, France, Belgium and Switzerland.

It is the earnest desire of the committee to secure all possible information bearing upon the subject, and it is hoped that parties interested will submit their views to the committee promptly. Sets of the circular letter and blank for replies will be supplied to any applicant by addressing the chairman, Room 21, Senate Annex, Washington, D. C.

The committee, which is composed of three members of each House, will probably assemble in Washington soon after the

middle of November for the purpose of formulating a report to Congress accompanied by the draft of a law which will place domestic industries on as favorable a basis as similar industries in foreign countries. During their sessions in Washington hearings will probably be given in order to supplement the information obtained through the interrogatories above set forth. Due notice of the time of such hearings will be given to the public.

OBITUARY NOTICE.

PROFESSOR AUGUST KEKULÉ'S part in the advancement of chemistry has been so important that his death on the 13th of last July has brought a feeling of sorrow to the hearts of chemists throughout the world.

Kekulé was born at Darmstadt, the birthplace of Liebig, on the 7th of September, 1829. It was the intention of his parents that he should become an architect, and he entered the University at Giessen as a student of architecture. He devoted himself with application to the studies bearing on his future calling, but like many another student who came within the range of Liebig's influence, he was filled with an enthusiasm for chemistry, which changed all his plans for the future, and led him to devote himself to this science. It is quite possible that his preliminary architectural studies had much to do with turning his mind toward the ideas of molecular structure or molecular architecture, which he subsequently developed. Kekulé also studied in Paris under Dumas, and in London under Williamson. In 1856 he became privatdocent at the University of Heidelberg. He was appointed professor of chemistry at the University of Ghent (Belgium) in 1858; and in 1865 was called to the University of Bonn, where he remained until his death.

Kekulé's first published work appeared in Liebig's *Annalen* for 1850. Four years later he published his second paper, in which he described thiacetic acid and discussed the action of phosphorus pentasulphide on oxygen acids.

The period from 1854 to 1874 was one of the greatest activity with Kekulé. Since 1874 he has made comparatively few con-

tributions to chemistry, although occasional papers have appeared. In spite of the great number of investigations he has made, chemistry is most indebted to Kekulé for his great generalizations and theoretical suggestions.

He extended Gerhardt's type theory by adding the marsh gas type and introducing the idea of mixed types. These types made clear to him the difference in the power of the elements to hold other atoms in combination, and he developed the idea of valence, first put forward by Frankland, so that this new property of the elements was at once recognized by chemists, the conception of atom-linking followed at once, and this made possible the transition from the type theory to our present conceptions in regard to the structure of compounds.

In this paper published in 1858 Kekulé says: "It is the substitution and relation of the atoms and not radicals, that we must look to in order to get a clearer idea of the nature of these compounds."

He closes this remarkable paper with the following words: "In conclusion I believe that I should emphasize that I do not set much value upon this kind of speculation. But since chemistry, in its entire lack of exact scientific principles, must content itself for the time with the most probable and useful theories; it appears proper to present these views, for they, as it seems to me, give a simple and entirely general expression for the latest discoveries, and because moreover their application may be the means of discovering new facts."

It is not too much to say that the ideas thus modestly put forward, supported by his subsequent work, were the prime cause which led to the abandoning of Gerhardt's types for our present structural formulas.

These ideas had made considerable progress, when in 1865 Kekulé published his now well known hypothesis in regard to the constitution of benzene. Seldom has a theory in chemistry been so suggestive or given rise to so much investigation as this benzene theory. The rich and manifest results accruing from these investigations testify sufficiently to the utility of the theory.

Many students of chemistry were attracted to Bonn; these Kekulé inspired with a love of investigation that has been

exceedingly fruitful for the science. Besides his work as a lecturer and investigator, he began in 1860 and finished in 1861 the first volume of his *Lehrbuch der organischen Chemie*, a book that was epoch-making with its new ideas and new methods of presenting this complex subject. The book was received with enthusiasm among chemists, and has served as a model for subsequent works in the same field. Three volumes of this work were finally published, but the work was never completed. He was also for many years one of the editors of Liebig's *Annalen*. During his last years he suffered much from ill health, having followed too literally Liebig's advice: "If you would become a chemist, you must ruin your health. He who does not ruin his health by hard study in these days comes to naught in chemistry."

In 1890 the German Chemical Society celebrated the twenty-fifth anniversary of Kekulé's benzene theory. The meeting was largely attended by chemists from all parts of the world. Addresses were given by A. W. Hofmann, the President of the Society, Adolph von Baeyer, Kekulé's oldest pupil, and by Kekulé himself. A full account of the meeting has been published.[1]

G. M. RICHARDSON.

OCT. 17, 1896.

NEW BOOKS.

MANUAL OF DETERMINATIVE MINERALOGY WITH AN INTRODUCTION ON BLOWPIPE ANALYSIS. By George J. Brush. Revised and Enlarged by Samuel L. Penfield. 14th Edition. pp. ix + 108. John Wiley & Sons. Price, $3.50.

This revision, with the exception of the tables, is practically a new book. The author states that "A complete revision of the tables for the determination of minerals will be made as soon as possible, and a short chapter on crystallography and the physical properties of minerals will be prepared, but until this work can be accomplished, use will be made of the tables and of the short introduction to them from the last edition of Professor Brush."

This proposed revision of von Kobell's table is greatly needed. When it is finished the book bids fair to be as nearly perfect as text-books can well be. The introductory chapter has been rewritten with evident care and by a practiced hand, and as it

1 *Ber. d. chem. Ges.*, 23, 1265.

now stands this edition is a great improvement over preceding ones.

"In preparing the introductory chapters, great pains have been taken in the selection of the tests for the elements. Many of them are performed by means of the blowpipe, but chemical tests in the wet way are recommended when it is believed that they are more decisive." To this evidence of good common sense it may be added that in several places the author shows a desire and ability to make his knowledge of practical value. This is shown, for example, under gold, where careful directions are given for the detection of gold in poor gold ores and the like, first by the use of mercury and then without mercury. E. H.

THE ELEMENTS OF CHEMISTRY. By PAUL C. FREER, PH.D. x+284 pp. Boston: ALLYN & BACON. 1895. Introductory price, $1.00.

One feature in particular makes this book especially worth noticing, and that is its outright recognition of the great importance of quantitative work in an elementary course in chemistry. The recognition has been a long time on the way, and its absence has been a great detriment to the chemical instruction in secondary schools.

It is also pleasant to find Professor Freer recognizing that certain so-called physical matters are best reviewed at the outset of such a course. Indeed it would seem as if some such matters which are taken up in the present work, rather late in the course, would better be considered earlier (the laws of Mariotte and Charles for instance).

The book cannot be used to advantage by an inadequately trained teacher, but will certainly be found valuable to the student teacher on account of its excellent collection of experiments which are carefully planned and digested.

JOSEPH TORREY, JR.

TABLES AND DIRECTIONS FOR QUALITATIVE CHEMICAL ANALYSIS. By M. M. PATTISON MUIR.

This little work is evidently intended to increase the possibilities of lecture table instruction in qualitative analysis. It consists of such brief statements of processes and methods as will enable the student to attend to what is going on on the lecture table without running the risk of losing material which ought to get into his note book. The analytical methods described are, for the most part, such as have stood the test of time and experience. JOSEPH TORREY, JR.

THE LIQUEFACTION OF GASES. Papers by MICHAEL FARADAY, F.R.S. (1823-1845). Alembic Club Reprints No. 12. 79 pp. Edinburgh: WM. F. CLAY. Price, two shillings.

In this little book of seventy-nine pages there is much matter

that will be of practical service to every one who teaches elementary chemistry. Its value to investigators and advanced students is sufficiently obvious. Students ought to be introduced to the classics of chemistry at a comparatively early stage of their development. They are not as a rule, at present, because the original papers are seldom accessible to the teacher. The publication of Ostwald's "Klassiker" was the first step in the right direction, but the fact that they are in German makes them inaccessible to many who most need them.

JOSEPH TORREY, JR.

CORRESPONDENCE.

POLARIZATION BY DOUBLE DILUTION.

UNITED STATES DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY,
WASHINGTON, D. C., Nov. 27, 1896.

Editor Journal of the American Chemical Society, Easton, Pa.:

DEAR SIR: By accident a portion of the rule for calculating polarizations by double dilution in our paper published in this Journal, 1896, Vol. 18, pages 428 to 433, was omitted.

Page 430, beginning at the end of line 9, the rule for the approximate calculation of results obtained by Scheibler's method of double dilution should have this addition after the words "small flask," "multiply the difference by two and subtract the product from the reading in the small flask." This is equivalent to multiplying the reading obtained from the solution in the large flask by four and subtracting the reading obtained from the solution in the small flask from the product. The result is the corrected reading and, when a solution of double the normal strength is polarized in a tube of double the normal length, must be divided by four to obtain the percentage. In this case a simpler and equivalent rule for calculation is the following: Subtract one-fourth the reading of the solution in the small flask from the reading in the large flask and the result will be the corrected percentage.

Page 430, end of line 17, the word sucrose should be lactose.

Page 432, the figures in the table in the column headed "Vol-

ume of precipitate," were calculated before the exact formula on page 430 was evolved, and are somewhat at variance with the results obtained by use of the formula. The formula gives the following numbers: 5.26, 10.71, 4.88, 9.86, 5.05, 5.41, 4.53, 4.12, 3.87, 4.99, 3.33, 4.22, 16.23. The numbers in the column headed "True volume in 100 cc. flask" must be changed accordingly.

Respectfully,

H. W. WILEY,
E. E. EWELL.

BOOKS RECEIVED.

Bulletin No. 33. Commercial Fertilizers and Chemicals, and Other Information in Regard to Fertilizers. Under the supervision of Hon. R. T. Nesbitt, Commissioner of Agriculture of the State of Georgia. Dr. George F. Payne, State Chemist. Atlanta, Ga.: George W. Harrison, State Printer.

Manual of Determinative Mineralogy, with an Introduction on Blowpipe Analysis. By George J. Brush. Revised and enlarged by Samuel L. Penfield. Fourteenth Edition. x+ 108 pp. New York: John Wiley & Sons. Price $3.50.

Jahrbuch der organischen Chemie. Herausgegaben von Gaetano Minunni. Palermo. Zweiter Jahrgang. 992 pp. 1894. Leipzig: Johann Ambrosius Barth. (Arthur Meiner). 1896.

A Brief Introduction to Qualitative Analysis; for Use in Instruction in Chemical Laboratories. By Ludwig Medicus. Translated from the Fourth and Fifth German Editions by John Marshall. Fourth Edition. Philadelphia: Printed by J. B. Lippincott Co. 1896. 203 pp. Price $1.50.

Bulletin No. 43. Second Series. Bovine Tuberculosis in North Louisiana. Bulletin of the Louisiana State Experiment Station, Baton Rouge, La. 1896. 20 pp.

ERRATUM.

On page 994 (November number), seventh line from bottom, instead of "extra internal pressure" read "extra external pressure."

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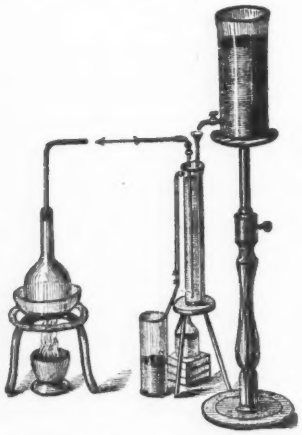

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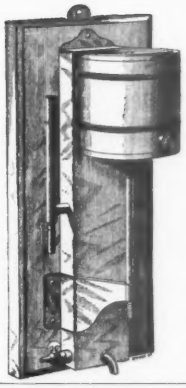

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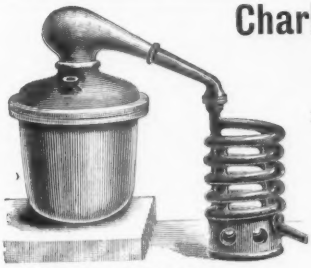

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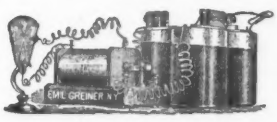

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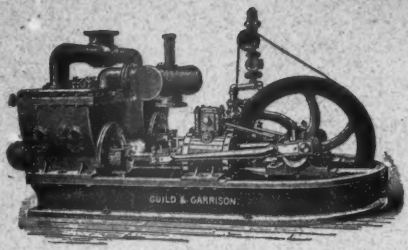

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